SOCIETY OF CHEMICAL INDUSTRY

FOUNDED 1881   ::   INCORPORATED BY ROYAL CHARTER 1907

PROCEEDINGS
OF THE
CHEMICAL ENGINEERING
GROUP.

Vols. 5 & 6a
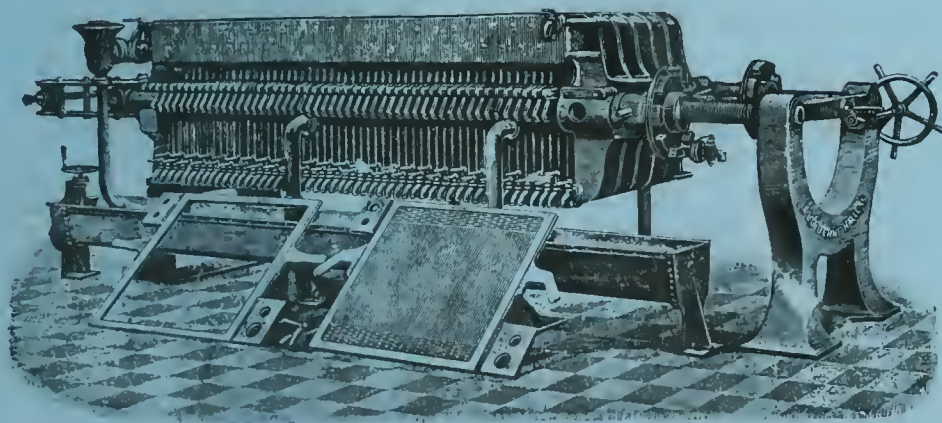
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SOCIETY OF CHEMICAL INDUSTRY

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J. ARTHUR REAVELL, M.I.Mech.E., M.I.Chem.E.

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CHEMICAL ENGINEERING GROUP

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Vols. V & VIa

1923–1924

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CHEMICAL ENGINEERING GROUP

Vols. V & VIa - 1923—1924

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The Fifth and Sixth (A)

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of the

CHEMICAL ENGINEERING GROUP OF

THE SOCIETY OF CHEMICAL INDUSTRY

1923—1924

The work of the Committee of the Chemical Engineering Group during the year ended December, 1923, was largely a continuation of the policy initiated in 1922 of taking all possible steps to strengthen the organisation of the Group and improve its financial position. It is satisfactory to report that the efforts made to this end have proved extremely satisfactory.

The work of the General Committee has proceeded more smoothly and efficiently with the increased number of members introduced after the Annual Meeting of 1922.

The Data Sheet Committee issued during 1923 three Data Sheets, Nos. 8, 9 and 9A respectively, giving various Air Drying data. The work necessary to produce these sheets was extremely laborious, and the data now set out in such convenient form have been in very great demand.

The relations between the Group and the Society of Chemical Industry continued to be most intimate and cordial. The Committee have pleasure in recording once more their appreciation of the assistance at all times willingly rendered by the President of the Society and the Hon. Treasurer in the discussion of doubtful points as and when they arose.

The organisers of the World Power Conference (1924) approached the Council of the Society of Chemical Industry with a request for assistance in the organisation of the Chemistry and Physics Section of the Conference, and the Council delegated this duty to the Committee of the Group. Ultimately a new section of the Conference was constituted—viz., "The Gas and Fuel" Section, the detail work of which was taken over by the Secretariat of the National Gas Council. The Committee are pleased to think that the efforts they made in bringing this new section into being resulted in making the World Power Conference a much more comprehensive activity than would have been possible had the subject of Power been treated from the Electrical side only. The Conference was held at the British Empire Exhibition, Wembley, from June 30th to July 12th, 1924 (Halls Nos. 1 and 2), and was attended by a very large and representative body of visitors from abroad. The Proceedings of the Conference are being separately issued and will constitute a unique Volume of Reference for engineers for many years to come.

The Society of Chemical Industry continues to collect the subscriptions of members of the Chemical Engineering Group, and this arrangement is working with increasing smoothness and efficiency. There is no question but that it is one of the most advantageous improvements in dealing with the Group's revenue that has so far been introduced.

The relation between the Group and the Institution of Chemical Engineers has been definitely settled, the Group being a tenant, at an agreed inclusive rental, of some of the Institution's office premises at Abbey House, Victoria Street, London, S.W. 1. A Joint Secretariat carried on the work under the general direction of the two Hon. Secretaries and with the immediate supervision of a Joint (paid) Assistant Secretary. Many of the members of the Group Committee are on the Council and/or Committees of the Institution, and it is no exaggeration, to say that the mutual assistance thus afforded has been of the greatest value to the organisations concerned.

The meetings arranged by the Committee during the year are set out hereunder.
1923.
January 16th.—Joint Meeting with the Hull Chemical and Engineering Society. Paper by J. Arthur Reavell, Esq., on "THE MANUFACTURE OF WOOD EXTRACT."

February 9th.—Meeting in London. Paper by Prof. F. G. Donnan on "SOME CONTROL FORMULÆ IN LEACHING AND EVAPORATION."

March 12th.—Meeting in London. Paper by B. Heastie, Esq., on "HEAT TRANSFERENCE."

This paper was of particular interest and importance and gave rise to a very helpful discussion.

November 16th.—Meeting in London. Paper by Prof. J. W. Hinchley on "A NEW SOURCE OF POTASH AND ITS INDUSTRIAL EXPLOITATION."

This paper, with manufacturing details, was given in a much fuller form before a joint meeting of the Group and the London Section of the Society on February 4th, 1924. (See below.)

December 12th.—Joint Meeting in Liverpool with the Liverpool Section of the Society of Chemical Industry. Paper by J. Brewis, Esq., on "VEGETABLE OIL EXTRACTION."

This paper was preceded by a visit to the African Oil Mills by arrangement with the Hon. Secretary of the Liverpool Section and by kind permission of the Mill Management.

No Conference was held during 1923.

The membership of the Group increased during 1923 from 315 (in 1922), to 386. There is no doubt that this is in part due to the new method of collecting subscriptions, using the Society's organisation for that purpose. As a consequence the year closed with a small credit balance.

The volume now issued includes, in addition to the papers (and discussion), above mentioned, the following contributions :—
1924.

February 4th.—Meeting in London. Paper by Prof. J. W. Hinchley on "THE MANUFACTURE OF POTASH AND OTHER SALTS FROM LEUCITE."

February 26th.—Meeting in London. Symposium on "THE TREATMENT OF WATER FOR INDUSTRIAL PURPOSES." Papers were contributed as under :—

"THE TREATMENT OF CONDENSING WATER," by H. W. Coulson.

"WATER PURIFICATION FOR INDUSTRIAL PURPOSES," by J. P. O'Callaghan.

"WATER-SOFTENING BY MEANS OF DOUCIL," by T. P. Hilditch and H. J. Wheaton.

"GENERAL METHODS OF WATER PURIFICATION FOR INDUSTRIAL PURPOSES," by B. Heastie.

This meeting was held jointly with the Institution of Mechanical Engineers.

March 4th.—Meeting in Hull. Paper by E. A. Alliott, Esq., on "CENTRIFUGAL DRYERS AND SEPARATORS—THEIR CONSTRUCTION AND USE."

March 7th.—Meeting in London. Papers by Major V. F. Gloag on "ACTIVATED CARBON," and Prof. E. C. Williams on "SILICA GEL AS AN INDUSTRIAL ADSORBENT."

March 28th.—Meeting in London. Paper by L. Andrews on "KINETIC ELUTRIATION."

April 4th.—Meeting in Birmingham. Paper by Capt. F. J. C. China on "THE PREMIER AND OTHER DISINTEGRATING MILLS AND THEIR APPLICATIONS IN INDUSTRY."

In accordance with a resolution of the General Committee the papers first cited include some of the very greatest interest.

The Committee regret to report the unfortunate death, during January, 1924, of the Assistant Secretary, Mr. A. C. Flint, who has performed the duties of that office almost from the inception of the Group. The Committee were able to signalise their appreciation of Mr. Flint's services by assisting his widow and family during the early days of their bereavement.

Following a period during which the secretarial work was carried out by voluntary help, the Committee, jointly with the Council of the Institution of Chemical Engineers, appointed Mr. C. J. T. Mackie to the vacant post of Assistant Secretary to the two organisations.

The Fifth Annual Meeting of the Group was held at the Engineers Club on May 2nd, 1924, and was a most interesting and enjoyable function. The Balance Sheet and Accounts were received and adopted at this meeting, and a report of the Committee's work (as summarised above) presented by the Hon. Secretary and adopted by the meeting.

The Committee are of opinion that the year 1923 was most successful in developing and consolidating the Group's influence, and they wish to express their appreciation of the constant support received from members of the Group, which enables this happy result to be recorded.

LIST OF OFFICERS AND COMMITTEE, 1923.

The Committee and Officers of the Chemical Engineering Group and the names of the Honorary Correspondents or Local Sectional Representatives for 1923 are given below :—

Chairman : J. Arthur Reavell. Esq.. M.I.Mech.E., M.I.Chem.E.
Vice - Chairman : C. S. Garland, Esq., B.Sc., A.R.C.Sc., F.I.C., M.I.Chem.E.

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Glasgow .. J. MacGregor, Esq., M.I.Chem.E.
Manchester H. Cheetham, Esq.
Liverpool .. W. E. Gibbs, Esq., D.Sc.

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W. H. Coleman, Esq., F.I.C.
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Glasgow J. MacGregor, Esq., M.I.Chem.E.
Manchester H. Cheetham, Esq.
Liverpool Prof. C. O. Bannister,*
W. Ramsay Sibbald, Esq., M.I.Chem.E.

* Since resigned.

THE MANUFACTURE OF WOOD EXTRACT.

By J. ARTHUR REAVELL, M.I.M.E., M.I.Chem.E.

In the following notes on the manufacture of wood extract, it is assumed that the extract is for use in the tanning industry, rather than for dyeing purposes. From the tanners' point of view the tan substances can be divided roughly into two classes, based on the fact that under the action of heat they yield either :—

1. Pyrogallol ;
2. Catechol.

Catechol tannins include : Birch-bark, oak-bark, Guebracho, mimosa-bark, mangrove, mallet-bark, hemlock, etc.

Pyrogallol tannins include, Myrabolan, valones, oak-wood, sumac, chestnut, algarobilla, babool, etc.

The practical difference between the two is that catechol tannins deposit reds and usually form very little acid. Pyrogallol tannins deposit ellagic acid, a whitish substance, known as " bloom," and give a fair amount of acid. On the whole they produce a rather softer leather than the catechol tannins. A lot of mixed tan extracts are manufactured, the idea being to use both the reds and the bloom.

Any substance extracted from the raw material other than tan is classed as a non-tan and includes sugar, glucose, nitrogenous and gummy matter. Non-tans play an important part in the process of tanning but do not come within the scope of this paper.

In passing, however, the list of tans given below showing the two classes and indicating their characteristics, will be of interest.

Having briefly reviewed the most commonly used extracts the actual process of manufacture must be considered, and in doing this it is important that the subject should be viewed as a whole. Fig. 2 will give a very good idea of the various stages in the process, the most important, of course, being the crushing or disintegration of the raw material in the mills, the extraction of the tan in the leaches and the concentration of the extract in the evaporating plant. It should be noted that the dust from the mills is collected and passes on to the mills ; also that the spent bark, in the case of some materials, can be used as fuel in the boilers or dried and sold as a by-product.

CATECHOL GLASS.

Material.	Tannin.	Non-tannin.	Insol.	Moisture.	District.	Notes.
Mangrove bark	33·09	10·60	43·80	12·50	Australia	The extract which is dark red in colour is rarely used alone.
,, ,,	22·17	16·76	61·07	—	India	
,, ,,	36·5	—	—	—	E. Africa	Best extraction temp. 80—90° C.
Mimosa bark ..	49·5	9·4	29·9	11·2	Australia	Sugar content of air dried bark = 43% of solid extract (20% moisture)—8·6%
,, ,, ..	41·4	7·9	39·2	11·5	,,	
,, ,, ..	40·1	13·0	36·8	10·1	S. Africa	
,, ,, ..	39·8	9·9	40·7	9·6	,,	
,, ,, ..	11·85	6·89	81·26	—	India	Best extraction temp., 70—80° C.
Extract	60·9	18·4	3·8	16·9	Natal	
Oak bark	10·9	4·2	74·7	10·2	—	One of the oldest materials used, not much favoured now owing to low tannin content. Best extraction temp., 70—80° C.
Guebracho	28·2	2·1	57·85	11·85	—	The Guebracho extract contains 63—67% tannin.
Solid extract ..	66·7	12·6	—	20·7	—	Best extraction temp., 80—90° C.

PYROGALLOL CLASS.

Material.	Tannin.	Non-tannin.	Insol.	Moisture.	District.	Notes.
Chestnut wood	11·2	3·4	—	—	—	Bark contains 4·8% sugars.
Bark ..	9·7	8·3	67·3	14·5	—	Best extraction temp., 90—95° C.
Myrobalans	33·0	13·1	41·7	12·0	—	Best temp. for extraction, 90—100° C.
Oak wood extract	31·5	8·5	—	60·0	—	Best temp. for extraction, 80—90° C.
Sumach	26·4	17·6	48·2	7·8	—	Ash and mineral 8·8%. The tannin is obtained from the ground leaf. Best extraction temp., 50—60° C.
Valonia	42·43	12·5	43·07	12·0	—	Contains 2% glucose. The tannin is obtained from the acorn " cup " and " beard."
Extract	68·0	24·3	0·2	7·5	—	Best extraction temp., 50—60° C.

PRELIMINARY TREATMENT OF RAW MATERIAL

Material such as bark or nuts must first be crushed before being dealt with in the leaches. This is usually done in a four-roll machine of standard design. For wood such as chestnut, Guebracho, etc., the logs are first cut into lengths of about three or four feet and are then put through a chipping machine, the chips being charged directly into the leaches.

There are, however, certain products, namely, some of the Indian barks, which do not contain any fibre. The leaching of these barks is a different problem and will be dealt with later, but at this stage it may be mentioned that it is necessary to grind these barks to a fine powder before they can be dealt with in the leaches. In the ordinary type leaches the dust from fibreless barks would block up the whole system. Apart from this a certain type of dust is produced from the ordinary grinding of barks, say mimosa, and it is very important that this should not be lost. In an extract factory recently samples of dust were collected and analysed, and it was found

arrange the crushers at a higher level than the leaches, so that the ground material can be dumped through chutes into the leaches. This meets the problem of mechanically handling the ground bark; but it is one of the points that must always be decided by local conditions.

Leaching.—The main objective in the process of leaching is to bring the water in contact with the tan-containing material in such a way that the whole, or as much as possible of the soluble tan is removed, and in doing this, to keep the labour involved down to the absolute minimum.

The perfect method of leaching is one in which the ground bark travels in one direction, while the liquor travels in the opposite direction, so that the fresh water enters where the spent tan leaves, and the liquor leaves where the fresh bark enters.

For many years, tanners have produced their own tan liquors, which they have used as thin liquor, and have generally extracted the tan from the bark by using their tan pits as leaches. The pits,
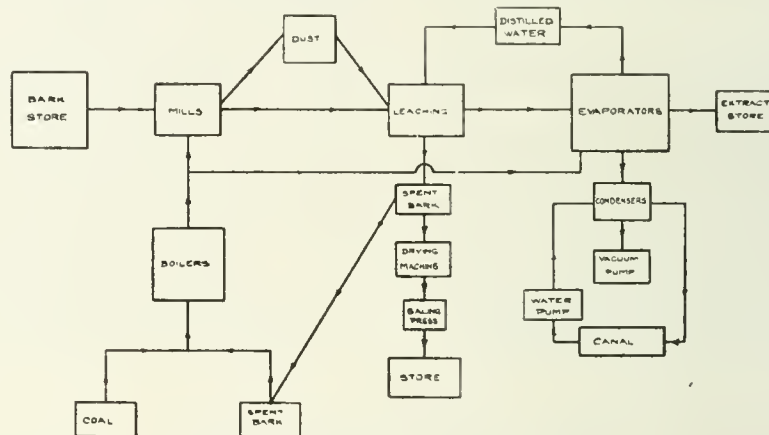
Tanning Extract Plant.   Flow Chart.

FIG. 1.

that they contained a much larger percentage of tan than the bark itself. Whereas the bark contained 33 per cent. tan, the dust contained 46 per cent. tan.

The importance of proper grinding of the different materials is shown by the following table :—

Percentage of Tan in Spent Bark with Varying Degrees of Grinding.

Calculated as a Percentage of the Spent Bark and of the same Moisture as the Original Charge.

Chopped bark (unground)	8·7% of tan
Closely ground bark	8·3% ,,
Medium ground bark	6·8% ,,
Finely ground bark	6·1% ,,

Conveyors.—After the bark has been crushed, it is necessary to convey it to the leaches, and for this purpose various types of conveyors have been adopted. While the raw material can be conveyed very easily in any ordinary type conveyor, it must be borne in mind that, under certain conditions, owing to the nature of the product, only copper or wood should be used in the construction of the apparatus.

The best plan is to take the bark, or other material, direct to the crushers by means of a conveyor and

however, were never designed for this purpose and it takes from four to six weeks to complete the extraction on account of the poor circulation. In addition the removal of the spent material from the pits is a difficult problem.

In designing leaches one of the most important points to bear in mind is accessibility, not only for charging and discharging, but also for cleaning out.

There are numerous types of leaching systems in use at the present time, these being :—

(1) The battery of open type, square or circular wooden vats.
(2) The battery of pressure autoclaves.
(3) The rotary system.
(4) The open trough containing an archimedian screw.
(5) The vacuum leaching system.
(6) Extraction in roller mills.
(7) Leaching in the Thornycroft extractor.
(8) The gas-agitation method for non-fibrous materials.

Wooden vats.—The normal wooden leach or vat has as its chief feature a perforated plate fitted about

12 inches from the bottom of the vat. This plate is usually of perforated copper so that the liquor, free from solids, can pass through into the space below.

The method of operating is, first of all to fill up to a few inches above the false bottom with water or liquor from another vat, then begin to charge with ground bark ; adding the rest of the water as the bark is thrown in.

In order to heat the liquor in the leach, a copper perforated steam coil is placed in the lower portion of the vat below the perforated plate. The steam is then turned on, the effect being to agitate, circulate, and at the same time heat the liquor.

The disadvantage of this arrangement is that the liquor is diluted to a considerable extent by the condensed steam, with the result that a greater amount of water has to be removed in the evaporators.

Very great improvements in design over the above described type of vat are shown in Fig. 2. It will be seen that the leaches are square, so that large discharging doors can easily be built into the sides at the minimum of expense. At the same time, the

Pressure Autoclaves

Pressure autoclaves, broadly speaking, are always used for the extraction of tan from wood, such as Quebracho and chestnut. The vessels are constructed of copper and gunmetal throughout. The filling doors are arranged on the top and emptying doors are either arranged on the side, or on the bottom, but an arrangement of perforated plate is fitted so that the liquor on being removed from the autoclave is taken away free from solid matter. The autoclaves are worked in rotation so as to approach as closely as possible to the ideal of counter-flow of wood and extract, as described later in the paper, the liquor being transferred from vessel to vessel through suitably arranged pipework by virtue of the pressure maintained.

The Rotary Leach

A battery of Rotary Leaching Vats consists of a series of drums operated in exactly the same way as the vats, with the exception that the mixing is obtained by the rotation of the drum instead of by
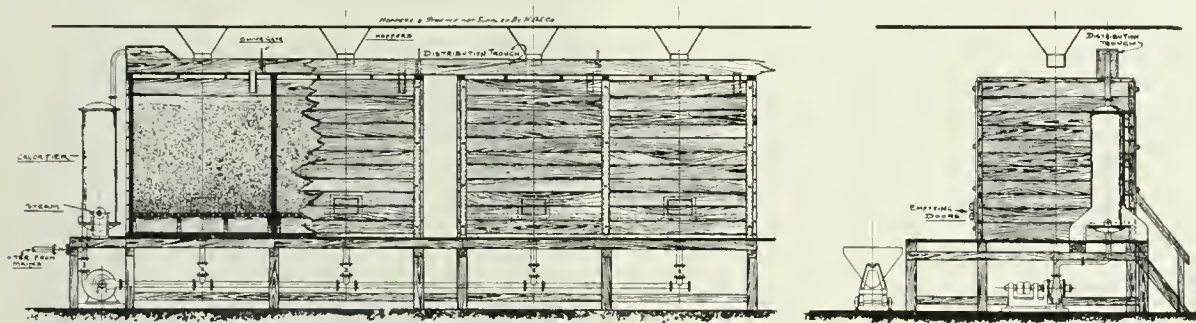
Arrangement of Leaching Plant.
Fig. 2.

maximum capacity is obtained in the minimum of space. Perforated coils are done away with, as heating in this way is not economical. All the heating is done by means of a calorifier, heated by boiler steam, and the condensate is used as the water supply to the leaches.

In order to circulate the liquors a centrifugal pump is used, so that there is positive circulation, as against thermic circulation where the open steam coil is used. The advantage of this arrangement in place of the method of blowing steam directly into the liquor will be better appreciated by taking a concrete case :—

A vat containing 9 tons of liquor to be raised in temperature from $20°$ C. to $90°$ C., or $68°$ F. to $194°$ F., in one hour, steam being supplied at 30 lb. pressure, through open coils.

Allowing 10 per cent. for losses, the steam required is roughly 1·35 tons : the steam condensed and added to the original quantity of liquor brings it up to $9 + 1·35 = 10·35$ tons ; or, to put it in other words, it dilutes the liquor by 15 per cent.

This added condensed water has to be re-evaporated in the evaporator, materially increasing the steam consumption of the whole plant.

the circulation of the liquor. In this way very thorough extraction is obtained.

The Open Trough System

This form of leach consists of a long wooden trough, in which is placed an archimedian screw built of copper and gunmetal. The new bark is fed in at one end and is carried forward by means of the screw, the water passing counter-current. This system has been tried in this country, but in spite of its theoretical advantages has been rejected, owing to various troubles, such as the jamming of the screw, the difficulty of removing the bark, and the unequal spending of the bark.

Leaching Under Vacuum

The Nance process of vacuum leaching has been successfully applied in South Africa, and very excellent results are claimed for it. The claims are based on the fact that by working under a very high vacuum, the cells which contain the tan burst, due to the vacuum, and the tannin can be extracted cold. This is in contradiction to the usual method, where high temperatures up to $100°$ C. are adopted. Nance

claims, and his results seem to show, that he obtains an extract practically free from insolubles, as the insolubles only come out at higher temperatures.

The plant used is practically the same as in the autoclave system, except that the vats are emptied and filled by means of vacuum instead of by pressure.

ROLLER MILL EXTRACTION

A proposal was made some years ago to adopt in tan extracting the ordinary triple rollers as used by sugar manufacturers for crushing sugar cane, and extracting the juice. A plant of this kind has been put up and seems to have given very fair results, but the difficulty is the very high cost of plant and the very heavy power consumption of the rollers.

THE THORNYCROFT EXTRACTOR

A method of extraction which has recently come to the fore was investigated and patented by Sir John Thornycroft. The machine used with this system was originally designed for the extraction of sugar from beet cossettes, for which purpose it proved so successful that its application to other processes was considered. Tests were carried out on an experimental machine under the directions of the writer on myrabolams and mimosa, as well as on some of the Indian barks. The extraction efficiency proved to be exceptionally high, and the advantage of the continuous operation obtained indicates that large savings of labour can be effected with this system.

The extractor is illustrated in Fig. 3, and it will be noted that it consists essentially of a vertical cylinder, which in the case of tan extract is in wood, while the metal work is in gunmetal. At the base of the cylinder there is a hydraulically operated plunger together with a system of valves, all automatically operated by a hydraulic pump through the medium of a timing valve designed so as to operate the plunger and valves in the correct sequence. The function of the plunger is to introduce the bark through an automatic seal into the base of the column at a definite rate, the bark being driven continuously upwards through the cylinder to the top, where it is discharged. The passage of the bark through the machine is assisted by an internal rotating screw. The water enters at the top and, passing through the extractor counter-current to the bark, leaves as weak liquor through a seal at the bottom.

In order to accelerate the extraction and at the same time heat the liquor, a series of centrifugal pumps (the number depending on the height of the column) are arranged at intervals on the outside of the cylinder, and circulate the liquor through external heaters.

A belt conveyor may be arranged to carry the spent bark, which leaves the machine continuously, away to the boilers, while the feed hopper can be placed directly under a chute from the chipping or grinding machines. The extractor then works absolutely automatically and produces a continuous stream of liquor, which may be delivered straight to the evaporator.

In the opinion of the writer the Thornycroft extractor is a very great advance on anything at present in use.

THE GAS AGITATION PROCESS

The large majority of the Indian barks are nonfibrous and are crushed with ease to a fine powder of about 200 mesh. Hence, it is impossible to leach them in the ordinary way. A system—namely, the gas agitation process invented by Mr. W. A. Fraymouth, has however been installed, and is now at work, which gets over this difficulty.

In this process there is a large central wetting vat surrounded by eight smaller vats called the agitators, as shown in Fig. 4. The incoming bark powder is delivered by a pneumatic conveyor through the cone into the top of the central vat, where it is thoroughly wetted by the stream of liquor issuing
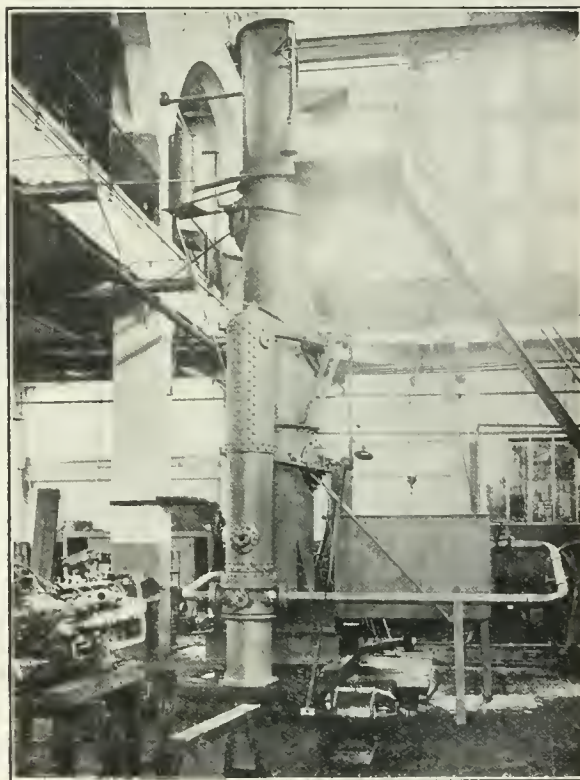
Fig. 3.

from the three vertical uplift tubes and return bends, B. in which continuous circulation is induced by the air-lift principle through the pipes, H. It is interesting to note here that the air lifts are operated by the exhaust of the gas engines supplying the power for the whole plant. The small amount of creosotic gases in the exhaust has the effect of stopping fermentation in the tanning liquors from the first moment of solution, with consequent saving of tannin.

The fourth uplift pipe return bend, B.S., is extended and is arranged so that it can be fitted into the top of any of the four vertical pipes, and thus discharge into any of the eight agitation vats. The underside of the bend is perforated at D to allow part of the liquor to run over a chute, E, into the annular zone, A,

formed by the vertical jute canvas veils, Q and V. The liquor passes slowly down the annular zone, J, upwards through the quiescent zone K, formed between the outer veil, V, and the side of the vat, to the launder, L, over which it passes as a clear liquor heavily loaded with tannin. The remainder of the liquor runs through into one of the agitators together with the tan stuff pulp.

Each of the eight agitators is arranged with a central uplift pipe of the same type as those in the central vat and also with a single jute canvas veil, R, forming a quiescent zone, V, between the veil and the sides of the vat. The eight agitators work in sequence as in the standard type of leaching system, the liquor travelling from vat to vat counter-current to the bark, and finally into the central vat. Continuous agitation is produced by the air lifts and the liquor passes through the quiescent zones and passes over the launders at the top into the next vat, the solids being deposited at the bottom. A pump, which can be attached to any one of the vats, is used to lift the strong liquor and deliver into the central wetting vat.

In practice, this system works extremely well and it has been tested on numerous Indian barks with excellent results. The efficiency is such that liquors from the central vat have been produced containing upwards of 20 per cent. solids.

Having reviewed the different leaching systems in most general use, the actual operation of a battery of leaches will now be gone into. It has been pointed out that leaching is carried out by means of counter-current working, that is to say, theoretically the ground bark is carried in one direction, and the liquid in another; but in actual practice, the bark remains in the vat, and the liquors are passed over it a given number of times. See Fig. 5.

Assuming there are six vats working normally, and that vat 4 is the one from which the spent bark is next to be removed, and the new charge of bark put in, the liquor is first drained out of 3, and taken to the evaporator, No. 3 being the last of the series which contains the fresh bark. Then 3 having had the liquor run off, the liquor from 2 is pumped into 3 (it can either be pumped through the calorifier or by-passed depending on its temperature).

The liquor from No. 1 is put into 2, No. 6 liquor into 1, No. 5 into 6, and 4 into 5, thus No. 4 is the leach emptied of its liquor and containing only bark that has had eight sets of water over it.

Having thoroughly drained the liquor from No. 4 the door is opened and the spent bark removed. The vat is then ready to receive its new charge of bark. A new charge of bark is put in, and the liquor pumped from 3 to 4, that is, over the new bark, from 2 to 3, 1 to 2, 6 to 1, and so on. No. 5 is pumped into 6, leaving 5 empty of liquor. Fresh water is now pumped into 5, and usually kept at a temperature of about 100° C. This is easily done by circulating through the calorifier.

Generally speaking, the last bark is kept at a high temperature, so that it can be thoroughly spent before being thrown out, and the temperature grows less towards the time when the liquor is meeting the fresh bark.

This gradual cooling of the liquor, while passing over the material, tends to give much clearer liquors as different soluble substances extracted at high temperatures gradually precipitate and are filtered out in the fresh bark.

Referring to the temperature in the final leach, that is, the leach containing the final bark, tanners reckon that 1 per cent. more tan is extracted by actually boiling in the last leach than by working at temperatures much below 100° C. It is, however, admitted that higher temperatures if used on fresh material destroy the tans.

A question that is always asked in regard to leaching is : What is the best shape and size for a leaching vat ?

Earlier in the paper it was mentioned that the tanners have used square leaching pits below ground,
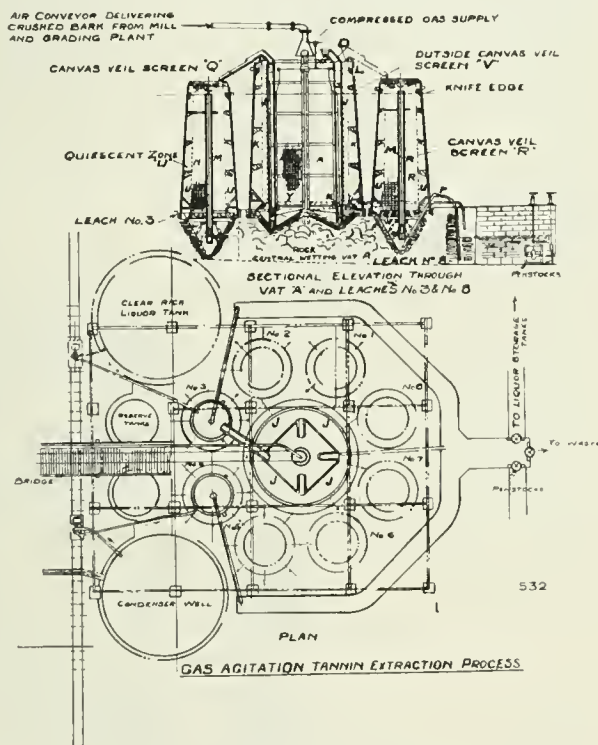
Gas Agitation Tannin Extraction Process.
Fig. 4.

the pits being about 9 ft. by 7 ft. and 6 ft. deep. They contained about 4 ft. depth of material. There is no doubt, however, that the best results are obtained from leaches placed above ground.

The size of the leach is determined very largely by the space available, and also the ultimate time of contact that is required to spend the bark. The minimum number of leaches that should be used is six, assuming the use of normal labour, although the writer knows of one successful system at work producing considerable quantities of extract with only three leaches. In this case, however, an exceptionally smart operator is employed, who takes an intense interest in the production of his liquors, and, in accordance with up-to-date practice, works on the intensive system, with a calorifier to heat his liquor.

The table below indicates the length of time the liquors are in contact with the bark and the quantity of bark dealt with in vats of various sizes. The figures apply to the leaching of mimosa and show

absolute maximum that can be obtained commercially from the bark ; but it should be borne in mind that manufacturers in this case produce their product with a view to obtaining the maximum amount of

TIME TABLE OF CONTACT.

		8		8		8
Number of vats	..		..		..	
Dimensions of vats ..	..	7′ 0″ × 7′ 0″ × 8′ 6″	..	8′ 0″ × 8′ 0″ × 8′ 0″	..	9′ 6″ × 9′ 6″ × 9′ 0″
Quantity of bark handled per 24 hours	..	15 tons	..	15 tons	..	15 tons
Quantity of water handled per 24 hours ..	..	48 ,,	..	48 ,,	..	48 ,,
Quantity of bark required per charge	..	2 ,,	..	2·8 ,,	..	4 ,,
Quantity of water required per charge	..	6·6 ,,	..	9·18 ,,	..	13·2 ,,
A vat is charged every	..	3·2 hours	..	4½ hours	..	6·4 hours
The liquor is in contact with the bark from	..	24·25 ,,	..	32·36 ,,	..	49·51 ,,

very clearly the advantage of the larger leaches in that with them ample time is available between the periods of charging the bark.

The quantity of liquor that can be produced per ton of bark and the density that can be reached depends on the method of leaching and also upon the amount of tan contained in the raw material. So many factors are involved that it is impossible to give figures for all materials. As an example, however, it may be taken that with reasonably efficient extraction 1 ton of mimosa bark yields 774/800 gallons of 45° barkometer liquor equivalent to about 9 per cent. tan by analysis, whilst 1 ton of mimosa and

saleable matter, whereas, the tanner, who manufactures for his own use, has other objects in view. He wants to get the purest extracts to use in the tanning of his leather, so that, whereas, the manufacturer claims to extract to under 2 per cent. of tan left in the spent material, the tanner seldom extracts down to 5 per cent. This 5 per cent. does not mean that with a bark containing 33 per cent. tan only 28 per cent. is extracted, as the 5 per cent. is calculated on the amount of tan remaining in the spent bark, and not in the original bark.

To put it more clearly, if 100 tons of bark are put into the leaches, and 45 tons of tan extracted, there
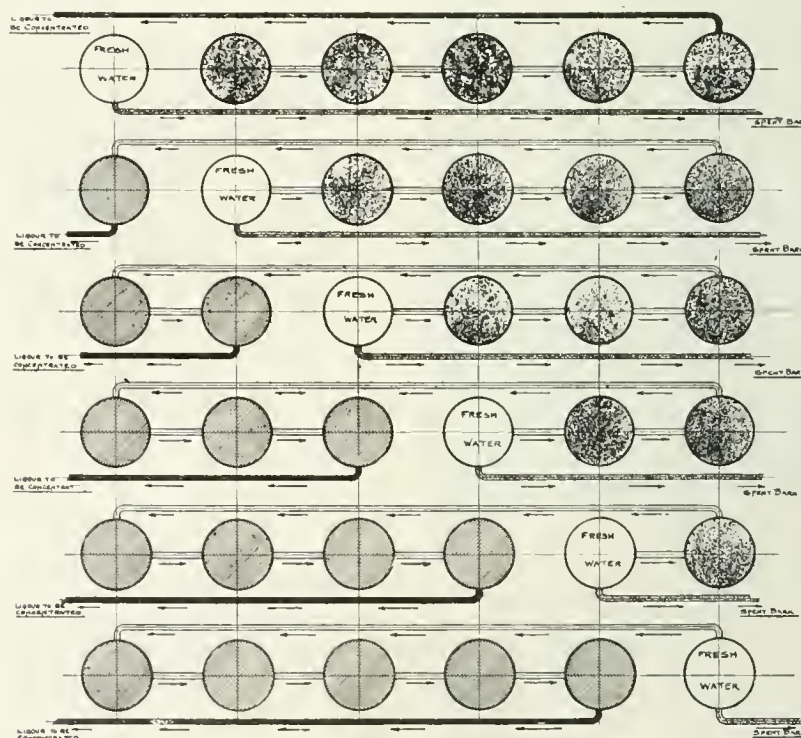
Diagram to show Rotation of Working.

FIG. 5.

myrabolan blend yields 900/1000 gallons of liquor of the same density as above.

The next question is the amount of tan left in the spent bark.

Extract manufacturers are much more particular than the tanners in this respect. They extract the

will only remain 55 tons of bark of the same moisture, and it is on this that the 5 per cent. is reckoned. It is equal to about 2·75 per cent. tan on the original bark.

At this stage it will no doubt be of interest to examine the curves on Fig. 6, one giving the relation between the density Twaddell, and the percentage of

tan extract, one based on mixed extract at 15° C., and the other based on pure mimosa extract at 15° C.

It will be seen that the relationship between the two is very close. For instance, at 35° Tw. the corresponding percentage of extract is in the case of mimosa 40 per cent. solids, and in the case of mixed extract 39 per cent. solids.

There are two other points of interest in regard to tan extract :—

1. The effect of heat in regard to acidity. With mimosa it is necessary to add acid to the liquor as it contains very little volatile acids, or sugar yielding material, or else to use myrabolan in conjunction therewith.

2. There is the question of the prevention of fermentation. In normal weather conditions $\frac{1}{2}$ per cent. of bisulphite of soda is used to prevent fermentation, but in the case of higher temperature this is not

Tan Extract (Mimosa).
$$y = 3.8x + .015x^2$$
$$x = \sqrt{66.67y + 16.044} - 126.7.$$
Fig. 6.
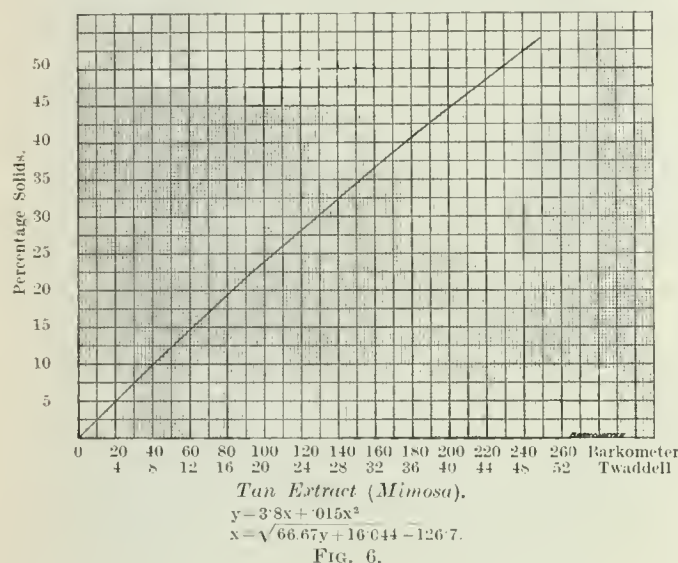
sufficient, as mimosa readily develops a yeast which ferments the sugars, and it is necessary to add $\frac{1}{2}$ per cent. cresylic acid.

Evaporation of Tan Extracts

In examining this phase of the process we will take as an example the case of mimosa extract. The liquor as it leaves the leaches is a thin liquid containing about 9 to 10 per cent. solids. If the extraction is good, it is as high as 10 per cent., but 9 per cent. can be taken as an average figure.

In order to concentrate this to produce the standard extract it is necessary to evaporate the water so that there is a content of about 38 to 40 per cent. solids in the concentrated liquid. This means an evaporation of about 78 to 80 per cent. of water. For instance, for 100 tons of liquid containing 9 per cent. solids, 80 tons of water have to be removed in order to give the 20 tons of liquor required at 40 per cent. solids.

Since the cost of steam is of considerable importance in the manufacture of tan extract, the problem that has to be considered is how to concentrate the liquor in the most economical way. The usual practice

is to use a triple effect evaporator. The principle of multiple effect evaporation is too well known to require description, but it may be mentioned that the type of evaporator shown is an example of the best modern practice. The chief features of this design are the arrangement of the heating surfaces, resulting in an extremely short time of passage of the liquor through the evaporator, and the construction of the separators whereby complete separation of liquor from vapour is effected by centrifugal force.

To take a concrete example of the importance of the question of steam consumption, assume that 50 barrels of extract (40 gallons each) are required at 40 per cent. solids : this is equivalent to, say, 10.5 tons of liquor at 180° bark. Assuming that the thin liquor contains 9 per cent. solids, it is necessary to evaporate 36.3 tons of water. In a triple effect evaporator, the steam consumption for this quantity will be 15 tons. Thus, 1 ton of 40 per cent. extract requires nearly $1\frac{1}{2}$ tons of steam.

It is of the utmost importance that all the condensate from the evaporator be returned to the leaching plant to be used for leaching, instead of using ordinary raw water. The advantages obtained in leaching with condensed steam as against raw water are clearly shown by the following example :—

With oak bark using distilled water 14.2 per cent. of tannin is recovered, as against only 6.9 per cent. where hard town's water is used. This gives a difference of 51.9 per cent. in tan extracted ; and Fig. 7 sums up by way of a heat balance the whole problem, and shows how all leaching and evaporation must be considered together if a success is to be made of the installation.

For 1 ton of mixed tanning material dealt with in the leaches, 857 lb. of steam are used in the calorifier, to heat the water and liquor for leaching, and 3180 lb. of steam in the evaporator. This latter comes back from the first effect and with it is taken the condensed vapour from the third effect consisting of 5300 lb. of water, making a total of 10,480 lb. of water to be used in the leaching.

This balance sheet shows that the steam from the boiler going to the evaporator and to the calorifier, together with the condensate from the condenser, just supplies sufficient water to do the necessary leaching. In this way, distilled water for the leaching is produced at a minimum cost. Further, the balance sheet has been based upon the leach liquor being delivered from the leaches at 9 Tw. or 11 per cent. solids, the barrelled liquor being at 40° Tw., and shows that 1 ton of coal is consumed for the production of 28 barrels of this particular blend of extract.

Referring to the previous figures discussed, based on the production of fifty barrels of 40 per cent. liquor, this it will be remembered necessitated 36.3 tons of water to be evaporated, equivalent in this case to about twenty barrels of extract per ton of coal.

The liquor from the triple effect is delivered at from 30 to 50 per cent. solids, depending on the use for which it is required. In installations abroad it is necessary to produce the extract in solid form, and there are two methods of carrying this out, both of which will be described.

The first is the use of a pan working under vacuum,

usually with a positive circulation arrangement which is extraneous to the pan itself. In some cases this circulation arrangement is fitted in the central tube of the pan. The difficulties of constructing this for thick liquor, which when carried to its final density is so thick that on drying it becomes brittle, must appeal to anyone with experience of vacuum pans, and it is absolutely essential, when a vacuum is used, to adopt some positive method of circulation. Even with positive circulation the amount of water evaporated from the liquor per sq. ft. of surface is very low indeed, so that a very large heating surface is necessary for the pan.

In this type of pan 3 lb. of water only are evaporated per sq. ft. of heating surface when working with a temperature difference of 50° C.

The cost of a vacuum installation of this type is very high, as, in addition to the large surface required

– FLOW SHEET –
SHEWING
WATER BALANCE BETWEEN EVAPORATION & LEACHING.
FIG. 7.

in the pan, and the expensive arrangement of circulating gear, all of which has to be constructed in copper, an air pump and condenser must be provided, and as the duty of the pan is much higher at the first part of the operation than in the final stages, the condensing plant must be large enough to condense the vapour given off when the pan is doing the maximum duty.

This type of plant is very rapidly being displaced by the Kestner Patent Evaporator, which works without a vacuum.

The evaporator is a modification of the well-known climbing film type, and consists of a calandria pro-

vided with long, vertical tubes through which the liquor passes at high velocity in the form of a thin film. The top and bottom boxes are arranged with division plates so disposed that the liquor travels in a serpentine manner through four sets of tubes in series, and finally is discharged with the water evaporated into the separator. The latter is of a special design and effectually separates the liquor from the vapour. The highly concentrated liquor leaves at the base of the separator, having a consistency approximately equivalent to that of thick treacle, and sets to a hard mass on cooling.

It is essential that the liquor is raised approximately to boiling point by being pumped through a heater (not shown in the illustration) before being fed to the evaporator, so that the latter has to perform its function of evaporation only.

The concentrated liquor leaving the separator contains only 8 to 15 per cent. of water, depending on the class of extract dealt with, and thus this evaporator performs the same duty as the pan without the complications of a condensing plant and mechanical stirring gear. It has been found that, owing to the very short time of contact of the liquor with the heating surfaces in an evaporator of this type, the temperature, although higher than that existing in a vacuum pan, does not in any way injure the product —in fact, experience has proved that there is less coloration produced than in the vacuum pan.

In some cases, when it is necessary to cool the extract quickly, or where the extract is to be packed directly in bags, a cooler extractor is used. This consists of a gunmetal casing provided with cooling water jackets and having a hopper into which the hot extract runs directly from the separator. The extract is forced through the cylindrical casing by means of a gear-driven worm, and is extruded through a nozzle in a semi-solid condition. The amount of cooling can be regulated, so that the extract can be run directly into bags without penetrating the canvas.

CRYSTAL EXTRACT

In some instances an extract known as crystal extract is required, and this is produced in a vacuum drier of the single drum type. The drier consists of a gunmetal steam-heated drum having a ground and polished surface and rotating on suitable bearings in an air-tight cast-iron casing.

A vacuum is maintained in the space between the drum and the casing, and the extract is fed on to the surface of the drum in the form of a thin film by means of a patented feeding device arranged along one side of the drum. As the drum rotates, the extract is dried and is removed by the scraper knives shown on the opposite side. The dry powder falls into a trough provided with a screw conveyor, which can rotate in either direction, and thus deliver to either of the containers placed at each end. When one container is full, the screw is reversed, and the second container starts filling. Meanwhile, the first is isolated from the casing by means of a special valve, the vacuum in it broken and the product discharged. In this way continuous working is obtained.

SOME CONTROL FORMULÆ IN LEACHING AND EVAPORATING.

With Special Reference to the Chilean Nitrate Industry.

By Prof. F. G. DONNAN, M.A., D.Sc., F.I.C., F.R.S., Hon. M.I.Chem.E.

The object of this note is to explain one or two simple formulæ which may be of use in controlling the working results of a plant where some valuable constituent (let us call it X) is extracted from the raw material by leaching, and isolated by concentration and crystallization of the leacher liquor. The Chilean Nitrate industry may be quoted as offering examples of such a type of plant, but many other cases will occur to the reader.

The first formula relates to the percentage extraction efficiency, E, of the leaching system.

Let C = percentage of X in the raw material.

R = percentage of X in the dry leached refuse.

Consider 100 parts of the raw material. The weight of X passing into the liquor is by definition $\dfrac{C\,E}{100}$, so that the weight of X left in the dry residue is $C - C\dfrac{E}{100}$. The weight of the total dry residue is $100 - C\dfrac{E}{100}$ and hence the weight of X in it is $\left(100 - C\dfrac{E}{100}\right)\dfrac{R}{100}$. Hence we get the "balance" equation :—

$$C - C\frac{E}{100} = \frac{R}{100}\left(100 - C\frac{E}{100}\right)$$

from which follows the formula :—

$$E = \frac{100\ (C-R)}{C} \cdot \frac{100}{100-R} \quad \dots\dots\dots\dots (1)$$

This formula adapts itself readily to the construction of a simple alignment chart from which the value of E can be read off when the values of C and R are known.

Equation (1) neglects, however, the possible moisture or combined water content of the raw material, and it also neglects any soluble substances other than X that may be extracted in the leaching operations. It is easy to introduce a correction term which takes care of the neglected quantities. Let w = percentage of moisture or combined water in the raw material, p = the amount of other soluble substances extracted, expressed as a percentage of the raw material. Putting s = p+w, the corrected formula is :—

$$E = \frac{100\ (C-R)}{C} \cdot \frac{100}{100-R} + \frac{100\ s}{C} \cdot \frac{R}{100-R} \cdot \cdot (2)$$

This equation can be readily deduced in the same manner as equation (1). For, starting with 100 parts of the raw material, and equating the two expressions for the weight of X in the dry refuse, we get

$$C - C\frac{E}{100} = \left(100 - C\frac{E}{100} - s\right)\frac{R}{100}$$

from which equation (2) follows at once. It is to be noted that C denotes the percentage of X in the undried raw material and that, in determining R, the representative sample of the leacher refuse must be dried at such a temperature that free and combined water are removed.

In a plant of the type indicated in the introductory remarks it will often be necessary to concentrate the leacher liquor by evaporation before it is run to the crystallisers. Frequently the cold mother liquor from the crystallisers and centrifugals will be returned as a feed liquor to the counter-current leaching system, if the leaching is carried out at such a temperature that the mother liquor possesses sufficient dissolving capacity. If not, the mother liquor must be returned to the evaporating system. The total wash water employed is balanced by :—

(1) The water going away with the moist drained leacher waste.

(2) The water evaporated in the concentration of the leacher liquor, the mother liquor, or both.

(3) The water evaporated by natural means in the crystallisers (and possibly also in launders and storage tanks.)

(4) The water removed in the drying of the crystals from the centrifugals (or draining shelves).

Of these quantities the important one is (2) as it involves the expenditure of heat and therefore of fuel (in so far as the waste heat in furnace gases or in the exhaust from non-condensing steam engines is not utilised). The amount of special evaporation required per ton of X extracted can be determined, in the case of any plant in actual operation, either by direct measurements on the evaporating plant, or indirectly by measurements of the total wash water used and of the quantities specified under (1), (3), and (4). It can also be calculated in another manner which may sometimes be more convenient, especially in the design of plant which is not yet in operation and in which the mother liquor returns as a feed liquor to the leaching system (as for example in the usual Chilean Nitrate practice).

Let r_e = ratio of water to X in the final leacher liquor.

r_c = ratio of water to X in the concentrated evaporator liquor.

r_m = ratio of water to X in the mother liquor from the crystallisers.

p = percentage of X in raw material.

e = extraction-efficiency of leaching system, expressed as a fraction of p

f = weight of X returning to the leaching system, per 100 tons of raw material treated.

w = weight of water removed (otherwise than by the tank waste and evaporation in evaporators) per ton of X extracted.

Then, per 100 tons raw material treated,

water in final leacher liquor = $(ep+f) r_e$,

water in final evaporator liquor = $(ep+f) r_c$.

Hence evaporation required, per 100 tons raw material = $(ep+f) (r_e-r_c)$

Water in returning mother liquor = $(ep+f)r_c - w\,e\,p$.

The amount of X extracted by this =

$$r_c(ep+f)\left(\frac{1}{r_e}-\frac{1}{r_m}\right) - w\,e\,p\left(\frac{1}{r_e}-\frac{1}{r_m}\right)$$

Net amount of fresh water entering leaching liquors = amount removed by special evaporation and by the other means specified above =

$$(ep+f)(r_e-r_c)+w\,e\,p$$

Amount of X extracted by this =

$$\frac{(ep+f)(r_e-r_c)}{r_e} + \frac{w\,e\,p}{r_e}$$

Since the amount of X extracted per 100 tons of raw material treated = ep, we have as the "balance" equation :—

$$r_c(ep+f)\left(\frac{1}{r_e}-\frac{1}{r_m}\right) - w\,e\,p\left(\frac{1}{r_e}-\frac{1}{r_m}\right) +$$

$$(ep+f)\left(1-\frac{r_c}{r_e}\right) + \frac{w\,e\,p}{r_e} = ep$$

This equation determines the value of f, and gives as a result :—

$$f = ep.\frac{r_c-w}{r_m-r_c}$$

Now the required evaporation per 100 tons of raw material treated = $(ep+f)(r_e-r_c)$.

Substituting for f its value from the preceding equation and calling Q_1 the required evaporation we obtain as the result

$$Q_1 = ep.\frac{r_m}{r_m-r_c}(r_e-r_c)\left(1-\frac{w}{r_m}\right) \ldots\ldots(3)$$

Denoting by Q_2 the required evaporation per ton of X extracted, $Q_2 = Q_1/ep$ and hence :—

$$Q_2 = \frac{r_m}{r_m-r_c}(r_e-r_c)\left(1-\frac{w}{r_m}\right)\ldots\ldots(4)$$

Determinations in the laboratory of the compositions of the leacher liquor, evaporator liquor, and mother liquor, combined with a determination of w from the plant data, enable Q_2 to be readily calculated from equation (4), which is of a form suitable for the construction of an alignment chart. If the mother liquor does not return to the leaching system but is sent to the evaporators, then the preceding equations reduce to the simple form :—

$$Q_1 = ep(r_e-w) \ \ldots \ \ldots \ \ldots \ \ldots \ (5)$$
$$Q_2 = r_e-w \qquad \ldots \ \ldots \ \ldots \ \ldots \ (6)$$

If we compare equations (4) and (6) and neglect the correction terms involving w, then, since

$$r_e > \frac{r_m}{r_m-r_c}(r_e-r_c) \ \text{if} \ r_m > r_c,$$

the evaporation required per ton of X extracted will be greater in the case where the mother liquor does not return to the leaching system, if the ratio of water to X for the mother liquor be greater than this ratio for the final leacher liquor, as will certainly be the case in a hot counter-current leaching system with return feed of mother liquor.

This conclusion involves the assumption that in both cases the value of r_c for the final leacher liquor is the same. This need not, however, be true. We may distinguish three principal cases :—

(a) Return feed of mother liquor to leachers. Concentration of leacher liquor by evaporation in evaporators before cooling and crystallisation.

(b) No return feed of mother liquor to leachers. Concentration of leacher liquor and of mother liquor in evaporators.

(c) No return feed of mother liquor. No concentration of leacher liquor by evaporation. Concentration of mother liquor in evaporators.

In case (c) the hot leaching is so conducted that a sufficiently concentrated leach liquor is obtained without evaporation. Denoting by Q the evaporation per ton of X extracted, and neglecting correction terms involving w, we may write :—

(a) $Q_A = \dfrac{r_m}{r_m - r_c} (r_e - r_c)$

(b) $Q_B = r_e$

(c) $Q_c = r_c$

where in each case r_c denotes the ratio water/X in the strong liquor going to the crystallisers, and r_e the ratio water/X in the final leach liquors which require evaporative concentration before cooling and crystallisation. If we compare cases (a) and (b) *for the same value of* r_e then, as shown previously, $Q_B > Q_A$ provided $r_m > r_e$. If we compare cases (a) and (c) *for the same value of* r_c, then $Q_c < Q_A$ provided $2r_m r_c < r_c^2 + r_m r_e$. In the foregoing discussion it has been assumed that no evaporation occurs in the leaching system. This will be true only if the leachers are closed. If open leaching tanks be employed and especially if the leaching liquors be heated, evaporation will occur during leaching. This will have the effect of allowing more wash water to enter the leaching system, but will not affect the formulæ given for Q_1 and Q_2 which refer to the special evaporation occurring in the evaporators. If, however, as has been for many years the practice in the Chilean nitrate industry, the liquors be strongly heated during leaching and finally boiled in the open leaching tanks, practically all the necessary evaporation may occur here. In this case, referring to equation (4), we have $r_e = r_c$ and therefore $Q_2 = 0$. In other words, no special evaporator is required, the leaching tanks themselves acting as evaporators as well as leachers, and the expenditure of heat for evaporation occurring here. We cannot in this case directly employ the equation (4) to calculate the evaporation occurring in the leaching tanks, though it may be used if we determine what the value of r_c would have been if, other things remaining equal, the *same* amount of leaching, but without evaporation, had occurred, and put r_c equal to the value for the final liquor actually obtained by combined leaching and evaporation in the tanks. This " fictive " value of r_c can be easily calculated if we know, per 100 tons of raw material treated,

(a) The amount of X leached out (i.e., ep).

(b) The amount of mother liquor returning to the leaching system.

(c) The amounts of water and X in this mother liquor.

(d) The net amount of wash water entering the leaching system (i.e., total wash water used less the amount removed with the tank waste).

But, as a matter of fact, there is no point in employing equation (4) in a case like this, for the determination specified sub (d) together with the determination of w give at once the evaporation occurring in the leaching tanks.

As this direct procedure can also be employed when evaporators are used, it may be inquired what practical use such an equation as (4) can possess. The answer is that, in the case of different plants and different " variants " of the leaching process, it will enable a central laboratory to calculate and check the varying amounts of evaporation required for specified or determined values of r_m, r_c and r_e (the value of w being small and usually fairly constant). It also enables the various factors determining the amount of evaporation required to be readily surveyed and their influence to be quantitatively determined in a simple and rapid manner.

Chemical Laboratories,
    University College, London.
9th February, 1923.

DISCUSSION.

PROF. DONNAN said his intention was to show them some elementary formulæ in regard to leaching developed in connexion with the Chilean Nitrate Industry. The first formulæ simply concerned leaching efficiency.

At the conclusion of the explanation of the formulæ PROF. DONNAN made a few remarks about the principles of leaching. He was quite clear that it should be perfectly easy to get a regular leaching efficiency of from 90 to 95 per cent., whereas a common value which he found in Chili, was an average of about 60 per cent.; the figures varied between 55 and 60 per cent.

THE CHAIRMAN said he was very interested in leaching, especially in regard to wood extracts etc., as distinct from nitrates, which had been dealt with by Prof. Donnan. He had some slides to exhibit, including one of the new Thorneycroft leaching apparatus, which had created some little stir lately. He then showed slides of apparatus, which is fully described on pp. 7 *et seq.* of this volume in the paper on " The Manufacture of Wood Extract."

DR. GORDON PARKER said that in the tanning industry a certain amount of formulæ was used, and they had gone a little bit further than the nitrate industry in that respect. The tanning industry certainly got a better yield in their extraction, and he did not think there was so much waste, but perhaps that was because the materials were more expensive. He had been very interested in some of Mr. Reavell's slides, especially the system of leaching he showed, and he would like to ask him why, in a system of leaching using batteries for bark or tanning

material, he pumped the material over from one leach to another. What objection had Mr. Reavell to what might be termed the old-fashioned press leaching system? Personally, he believed very strongly in that system, because he liked not to do the work, and the press leach system saved steam and power. Therefore, he had been rather surprised at Mr. Reavell showing a pumping system, which he (Dr. Parker) looked upon, no doubt in his ignorance, as somewhat antiquated.

Dr. W. R. Ormandy said he knew very little about leaching, and his only object in speaking was to mention a very peculiar method of extracting a certain amount of soluble material from vegetable matter in Germany which was being worked on a small scale and, he believed, very profitably. The ordinary method used for getting sugar out of sugar beet, was the diffusion battery, a series of copper vessels through which water gradually percolated, because sugar was contained in small cells inside the substance of the sugar beet. Count Schwerin, in Germany, however, found that by putting sugar beet between porous plates and forming an anode and a cathode, and passing an electric current through, cataphoresis took place, the water being pushed through the cells and coming out on the other side. It actually appeared to penetrate the cell wall and displaced the strong sugar solution inside the cell, at the same time displacing a large percentage of albumen and other materials. The system was worked on a large experimental scale, and it gave a very wonderful yield of a much purer sugar solution in a very concentrated form. However, the actual cost of the apparatus and the current was too great to render it possible as a commercial operation. The ordinary cost of leaching was comparatively small after allowing for the first cost of the diffusion batteries. The same principle was applied in Germany during the war to the removal of the bitter principle present in lupins, and the method had also been applied to the extraction of certain alkaloids which were thus got out in a much purer form from certain organic leaves and material.

Professor Hinchley mentioned a point in connexion with leaching efficiency which he regarded as important. Even with the best plant

it might pay to throw a portion of the material away and not aim at a higher efficiency, say, than 60 per cent. It very often did pay to throw away material, and in this particular case an efficiency of 90 to 95 per cent. might be purchased at too high a figure. The aim should be to increase the commercial efficiency.

Mr. Gardner asked Prof. Donnan if in any of the Chili plants they used anything in the nature of a heat exchanger by which the crystallising liquor was cooled and, at the same time, the extracting liquor was warmed. It seemed rather an obvious thing to do.

The Chairman, replying to Dr. Parker about the press leach, said the reason he pumped over in the system shown was with the intention of getting a definite and exact temperature in any leach at any time. That could be done with pumping, but it could not be done with the press system. In the system he had shown it was possible to get the exact temperature required at any time by means of the calorifier. If circulators were used, as was often the case, steam being used direct in the circulators, that added to the amount of water which had to be evaporated subsequently, and such good results were not obtained. Mr. Reid had raised the question of the density of leaching with the Thorneycroft system. Tests on beet sugar showed that a little higher density was obtained than when working with the ordinary batteries of autoclaves.

Prof. Donnan, replying first to Mr. Gardner as to the use of heat exchangers in the Chili plants, said the liquor which ran to the crystallisers was heated and about 10 per cent. of steam would be used in that way. It was a question of whether it was worth while doing it. Personally, he should say it would, as the cost of all fuel was heavy out there, and he saw no great difficulty in doing it.

The Chairman, referring to the cost of the Thorneycroft plant which had been referred to by Mr. Reid, said that it could be built in wood and not of copper, so that the expense need not be so enormous.

On the motion of Mr. Parrish a hearty vote of thanks was accorded both to Prof. Donnan and the Chairman.

HEAT TRANSMISSION IN COOLERS, HEATERS, AND CONDENSERS.

By BASIL HEASTIE, B.Sc., Assoc.M.Inst.C.E.

Paper read at a meeting of the Chemical Engineering Group on Monday, March 9, at the Chemical Industry Club. The Chairman of the Group, J. Arthur Reavell, Esq., in the Chair.

Among the many problems which confront the chemical engineer, that of heat exchange between gases or liquids in coolers, heaters, and condensers is a common one, and the amount of reliable data available is extremely small.

The object of this paper is to give a brief review of our present very incomplete knowledge of the laws governing heat transfer.

The flow of heat takes place in three distinct ways :—

(a) *Conduction* : in which heat passes from one point to another without appreciable displacement of the material particles ;

(b) *Convection* : in which the heat transfer is due to the displacement of the particles through a difference in density caused by the heat ;

(c) *Radiation* : in which a hot body gives out radiant energy.

In tubular heaters and coolers the flow of heat takes place mainly by conduction and convection.

The flow of heat by conduction can be expressed in a similar way to that of Ohm's law for the flow of electricity.

If we consider the flow of heat from (say) saturated steam to a liquid through a metallic plate, there are three main resistances :—

(1) A film of condensed steam on one face of a plate ;

(2) The resistance of the metal plate ;

(3) A stationary film of water on the other face of the plate.

In addition to these resistances, there may be surface resistances due to corrosion.

If h_1, h_2, h_3 denote the conductivities of the three obstructions to flow, the total heat flow per unit surface is equal to the total temperature drop :—

Total heat flowing (per unit surface)

$$Q = \frac{\text{Total temperature drop} \ (\Theta)}{\dfrac{1}{h_1} + \dfrac{1}{h_2} + \dfrac{1}{h_3}}$$

Thus $\dfrac{Q}{\Theta}$ or $H = \dfrac{1}{\dfrac{1}{h_1} + \dfrac{1}{h_2} + \dfrac{1}{h_3}}$

where H is the heat flow per unit surface for 1° temperature difference.

The point in question is explained in Fig. 1, which illustrates the heat exchange which takes place in a tubular cooler.

It will be realised that the temperature drop is chiefly due to the resistance of the two films and that the drop across the metal plate is practically negligible.

When a liquid or gas is in contact with a solid, such as the dividing walls of a tube, it has been
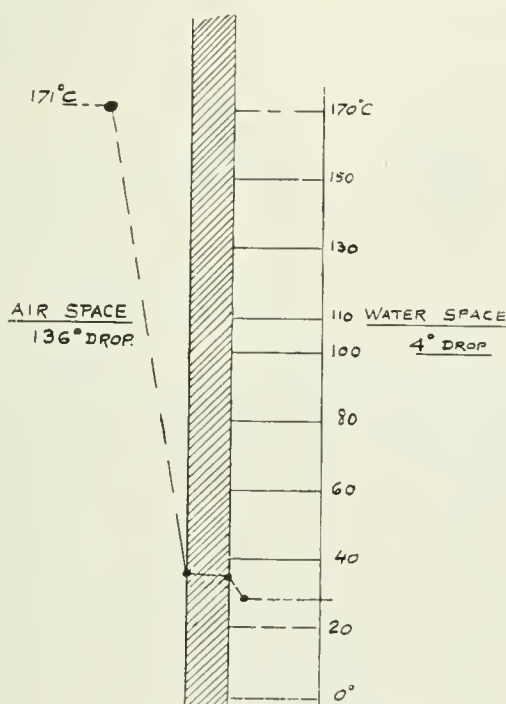
Temperature gradient.

Experiment.	Water mass flow. lb. ft.²/sec.	Air mass flow. lb. ft.²/sec.	Calories per m²/hr. 1° C.
1	20·5	2·4 V = 48ft./sec.	49
2	98	17·45 V = 347 ft./sec.	220

$S = \dfrac{48}{d''}$	Diameter of tube.	Air mass flow. 8 lb. ft.²/sec.	Calories per m²/hr. 1° C.
24·4	1·968″	,,	115
46	1·04″	,,	123
70	0·684″	,,	132

Cooling air in a tubular cooler.

FIG. 1.

shown that a stationary film of fluid adheres to the face of the solid, and that this film becomes thinner as the velocity of the fluid increases

B 2

and may break away from the solid body at the highest velocities.

It may be noted that the thickness of the film is dependent on the viscosity of the fluid.

Experiments by McAdams and Frost on the heat transmission between steam and water and benzine vapour and water are illustrated in Fig. 2. It will be realised that the resistance of
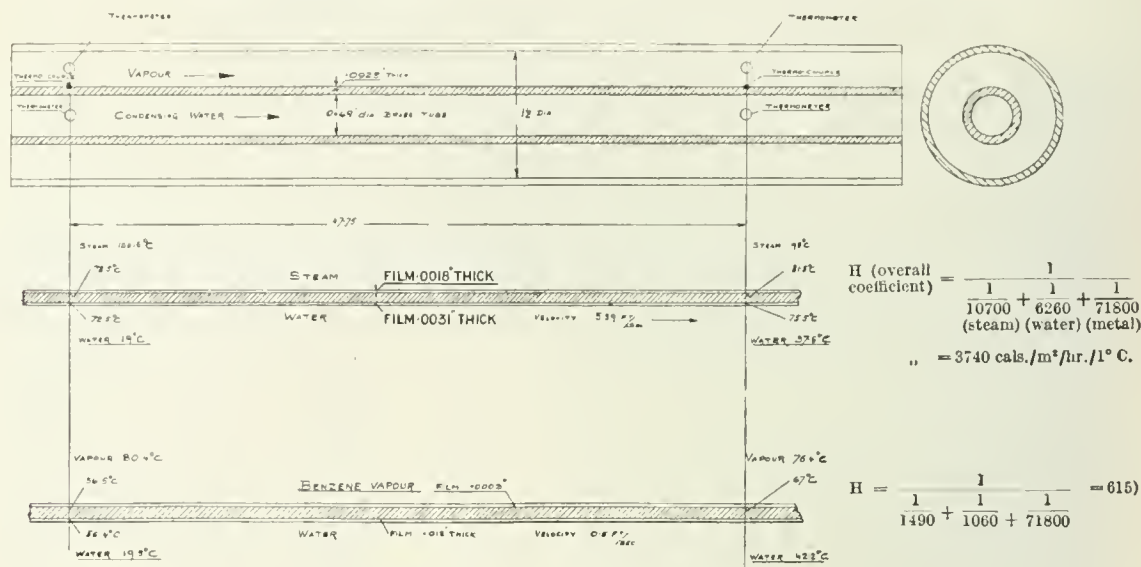
Film coefficients for benzine, steam and water.
Deduced from experiments made by Messrs. McAdam and Frost.

FIG. 2.

Through this film heat can be transmitted by conduction only, but as soon as the heat has passed through the film, convection becomes possible, the hot molecules are absorbed by the main body of the liquid and the resistance to heat flow becomes small.

Since most gases and liquids are very poor conductors of heat, a great resistance to heat flow occurs at their boundary surfaces, so that a large temperature drop takes place at those places.

It will be obvious, therefore, that in questions of heat exchange, turbulence should be promoted as much as possible.

The figures given in Fig. 1 are taken from experiments made some years ago by Jordan, in which heated air was passed through tubes, being cooled by water on the outside.

It will be noted that the temperature drop on the water side of the tube is small, so that there would be very little gained by increasing the water velocity.

The presence of incrustations or deposits on the metal surface may increase the resistance to the heat 100-fold, but it has been found that in the case of viscous liquids, the film resistances have been often so high that a certain amount of scaling does not affect the heat transmission to the extent one might expect. For instance, in 1921, Frost and Manley found that the heat transmission in 2-in. standard steel pipe was reduced by 25% after two months' use.

the films will be directly proportional to the thickness, inversely proportional to the conductivity; and the thickness will depend upon some functions of the viscosity and also upon the shape and position of the dividing wall.

Condensing vapour.

A formula for determining the co-efficient of heat transfer from vapour to a metal wall has been devised by McAdams and Frost,

$$h = 260,000 \frac{K}{N}$$

where K is the thermal conductivity in c.g.s. units.

N is the absolute viscosity in c.g.s. units.

Weber has given an equation for the determination of K:

$$K = 0.00359 \times S \, C \sqrt[3]{\frac{S}{M}}$$

where K is the thermal conductivity in calories per second per c.c.

M is the molecular weight.

S „ „ specific gravity.

C „ „ specific heat.

This equation has been put in the form of an alinement chart in Fig. 3.

It will be noticed that the McAdams-Frost equation takes no account of the velocity of flow.

The presence of air or incondensable gas in the steam or vapour affects the heat transmission considerably.

Robinson has derived a formula, which is shown in Fig. 4, by which the heat transmission coefficient for steam containing air may be determined. This formula should be accepted

is 0·08 in., the resistance of the metal is $\frac{1}{275}$th that of the film. If steel be employed in place of copper, the resistance is increased by about $\frac{1}{17}$th ; whilst if a lead tube is used, the resistance

C   S   K   M
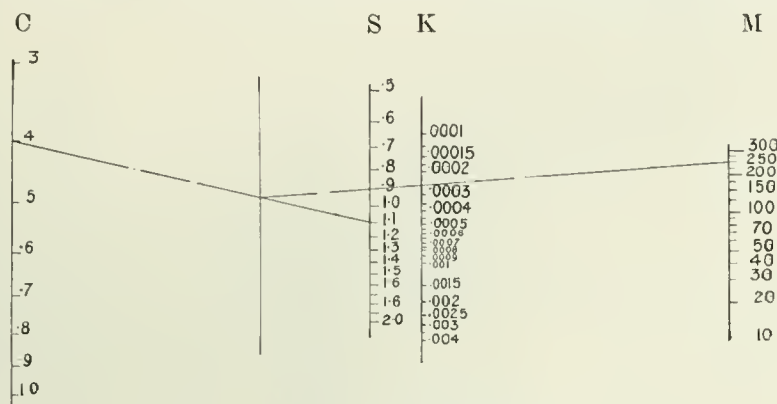
Thermal conductivity of liquids.

FIG. 3.

with caution, because it is very difficult to measure the amount of air present and in practice the air is not equally distributed. More research work is desirable before reliable figures may be accepted.
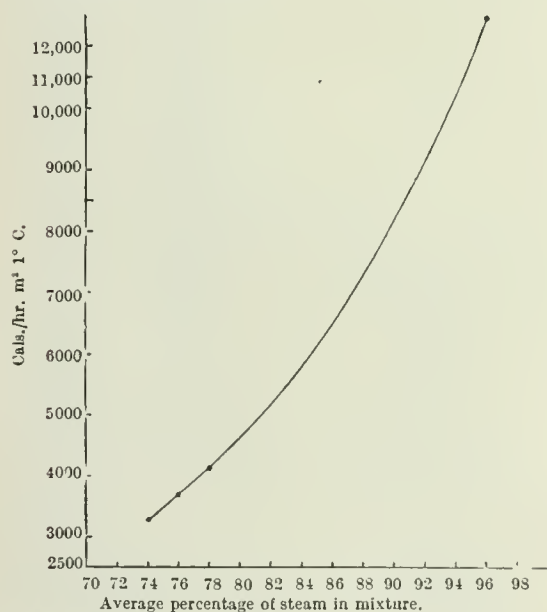
Heat transmission coefficient. Steam containing air.

FIG. 4.

Transfer through the metal wall.

In practice the resistance presented by the metal wall is neglected, unless the film resistances are very small, as in high-velocity heaters.

A table of conductivities is very useful and is given below. From this it will be seen that a film of water 0·003 in. thick is equivalent in resisting heat flow to a copper wall $2\frac{1}{4}$ in., or assuming that the average tube-wall thickness

Relative heat conductivity.

	Grm. Cals. per cm²., per cm, thick, per 1°C. drop per second.	Comparative figures Copper = 1000
Copper	1·041	1000·0
Iron	0·167	160·0
Calcium carbonate	0·005	5·0
Silica	0·0025	2·5
Water (not in motion)	0·0014	1·4
Air	0·000056	0·056

	Kg. Calories per m²., thickness 1 mm. per hour, 1°C. drop.	
Copper	374,000	
Iron	60,000	
Calcium carbonate	1,870	
Silica	935	
Water	520	
Air	21	

	Theor. kg. steam condensed per m²., thickness 1 mm. per hour, 1°C.	If the thickness is increased to 2 mm.
Copper	700·0	350·0
Iron	112·0	56·0
Calcium carbonate	3·5	1·75
Silica	1·75	0·87
Water	0·98	0·49
Air	0·040	0·020

is increased to $\frac{1}{25}$th ; and since for mechanical reasons it would be necessary to increase the tube thickness where lead pipes are used, the resistance would actually be much greater.

In the heat exchange between two highly viscous liquids, the resistance of the two liquid films due to their greater viscosity would determine the rate of heat flow and the material of which the tubes are made would have practically no effect.

Liquor film coefficient.

A number of formulæ have been derived by different investigators for determining this co-efficient, among whom may be mentioned Osborne Reynolds, Dalby, Hinchley, Porter, and Jordan in England, Boussinesq in France, Grober in Germany, Manley, McAdams and Frost in America.

Frost has recently suggested a simplified form of the Boussinesq equation, after having tested it practically using water and light oil :—

$$h = 714800 \ \frac{K}{D^{0·2}} \left(\frac{V\theta}{z} \right)^{0·8} \quad \dots \dots \dots \dots (1)$$

where K = coefficient of heat conductivity in c.g.s. units.

D = diam. of pipe in inches (internal).

V = velocity of flow in ft./sec.

z = specific viscosity of liquid (water at 20° C. = 1).

θ = density of fluid in lb./ft.^{3}

h = heat transmitted kg.-calories per m^2/ per hour/1° C.

The units adopted are a mixture of the metric and British systems and were adopted for convenience in comparing results from different sources.

In January, 1922, McAdams and Frost gave a full description of a series of tests on steam, water, benzine, and carbon tetrachloride vapour and suggested another formula :—

$$h = (\text{constant}) \left(\frac{K}{z}\right) \frac{V^{0.76}}{D^{0.17}} \quad \ldots \ldots (2)$$

The tests all agreed within less than 10% of the values obtained by the above equation.

This equation is given in the form of an alinement chart, which is shown in Fig. 5.

Fluidity of various liquids at different temperatures.

Liquid.				Temp. °C.		Fluidity.		K × 10⁴
Benzol	..	..	..	10	..	1·34	..	3·33
,,	..	..	..	20	..	1·55		
,,	..	..	..	30	..	1·735		
,,	..	..	..	40	..	2·03		
,,	..	..	..	50	..	2·31		
,,	..	..	..	60	..	2·57		
Aniline	..	..	..	12	..	0·166	..	4·08
,,	..	..	..	20	..	0·224		
,,	..	..	..	30	..	0·31		
,,	..	..	..	40	..	0·408		
,,	..	..	..	50	..	0·52		
,,	..	..	..	60	..	0·643		
Ethyl alcohol	..	..	..	10	..	0·656	..	4·6
,,	,,	..	..	20	..	0·795		
,,	,,	..	..	30	..	0·985		
,,	,,	..	..	40	..	2·17		
,,	,,	..	..	50	..	1·4	..	3·7
Petroleum	..	..	..	17·5	..	0·526		
Sulphuric acid	..	..	..	11·2	..	0·03		
,,	..	..	..	20	..	0·045	..	13
Rape oil	..	..	..	0	..	0·0094		
,,	,,	..	..	6·5	..	0·00194		
,,	,,	..	..	12·4	..	0·00325		
,,	,,	..	..	13·9	..	0·00355		
,,	,,	..	..	18·1	..	0·00591		
,,	,,	..	..	27	..	0·00835		
,,	,,	..	..	29·5	..	0·0104		
,,	,,	..	..	31·6	..	0·0111		
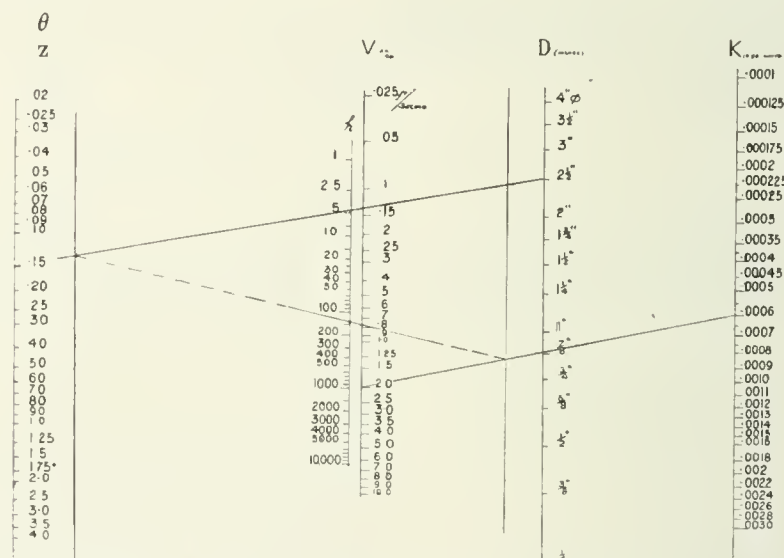
Heat transmission co-efficient (metric units), through a liquid film to a metal wall.

FIG. 5.

Prof. Porter has shown from theoretical considerations that

$$h \propto \frac{K}{D^{(1-n)}} \left(\frac{V\theta}{z}\right)^n$$

in which n = 0·75 has been determined from experiments by a number of investigators. This equation agrees very closely with the Frost equation (1).

McAdams and Frost found that the average value of h for steam (taken on 7 runs) was 11,600 calories/m.^{2} hour /1° C., whilst for water h was 1130 calories, the water velocity varying from 2·25 to 0·9 metres/sec. In view of the close results obtained from equation (1) it may be adopted with a certain amount of confidence.

Liquid.				Temp. C°.		Fluidity.		K × 10⁴
Mercury	..	..	..	0	..	0·59	..	148
,,	..	..	..	10	..	0·613		
,,	..	..	..	99	..	0·818		
,,	..	..	..	124	..	0·867		
,,	..	..	..	154	..	0·916		
Glycerin	..	..	..	18·3	..	0·00093	..	7·5
,,	..	..	..	20·9	..	0·0013		
Olive oil	..	..	..	15	..	0·01015	..	3·9
Caustic soda 4%	..	..	18	..	0·8			
,, ,, 8%	..	..	18	..	0·65			
,, ,, (normal solution)	..	25	..	0·81				
Water	..	..	..	20	..	$1·0 = \frac{1}{Z}$		

Gases flowing through tubes.

A considerable amount of work has been carried out by Nusselt, Grober, Weber, Dalby, Jordan and others. Particular mention should be made of a paper by Dalby in 1909, which was
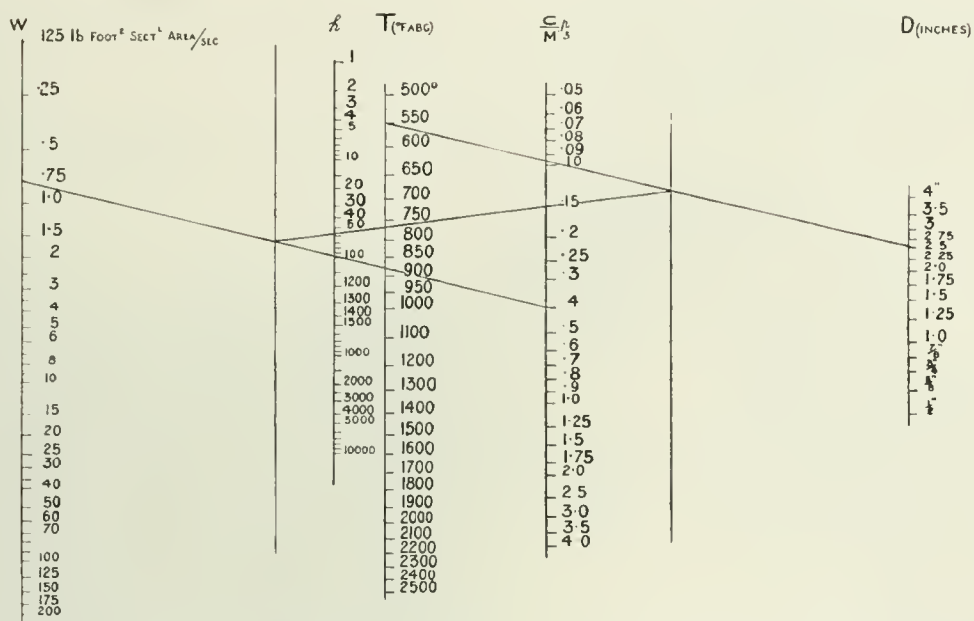
Heat transmission coefficient (metric units).   Gas film—flowing in circular pipes.

$$h \text{ (metric)} = \frac{4 \cdot 3 W^{0 \cdot 8} C p S^{0 \cdot 2} T^{0 \cdot 5}}{\text{Mol. wt.}^{0 \cdot 3}}$$

Fig. 6.

Approx. values of Cp.

Air	0·238
Oxygen	0·218
Hydrogen	3·406
Superheated steam	0 37—0·43
Carbon monoxide	0·245
Carbon dioxide	0·216

Cp = Specific heat at constant, p.
M = Molecular wt. of gas.
$\dfrac{Cp}{M^{0 \cdot 3}}$ = Factor for use in Weber's equation.

See Chart Fig. 6.
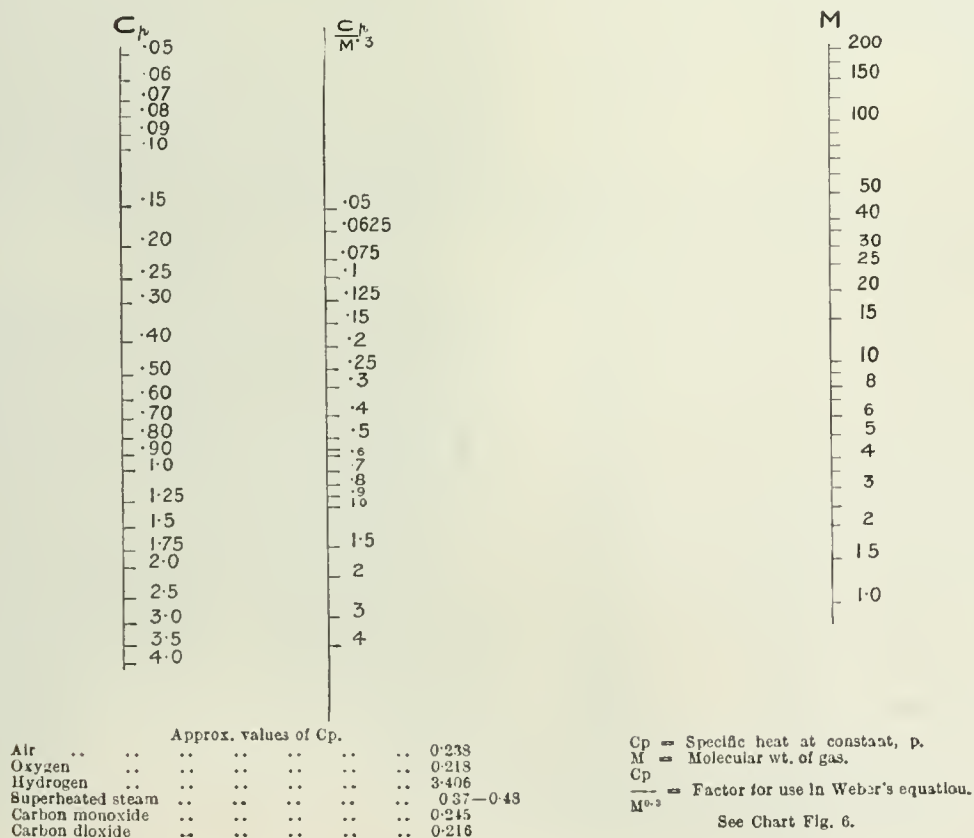
Fig. 7.

the outcome of two years' research—over 500 abstracts being consulted.

Probably the most accurate equation is due to Weber :—

$$h = \frac{4 \cdot 3 \; w^{0 \cdot 76} C_p S^{0 \cdot 2} T^{0 \cdot 5}}{(Mol. \; wt.)^{0 \cdot 3}} \quad \ldots\ldots\ldots\ldots\ldots (3)$$

where w = lb. per sq. ft. sectional area.

C_p = the specific heat at constant pressure.

S = the reciprocal of the hydraulic mean depth in ft. (in circular pipes S = 48/d), where d is the diameter of pipe in inches.

T = absolute temperature °F.

M = molecular weight.

An alinement chart (Fig. 6) affords a ready means of calculating this co-efficient.

In this equation the factor K is involved in (molecular weight)$^{0 \cdot 3}$ C_p. and mass flow W.

Fig. 7 is a convenient alinement chart for calculating $\left(\dfrac{C_p}{M^{0 \cdot 3}} \right)$

In addition transfer between liquids the transmission coefficient is higher when working at higher temperature ranges, but with the same temperature difference. This is due to the lower viscosity and higher conductivity with temperature increase. In the case of gases, however, the viscosity increases with increasing temperatures, e.g. :—

			0° C.	100° C.	300° C.	500° C.
Oxygen	..	..	1·84	2·34	3·19	3·89
Air	..	..	1·69	2·15	2·89	3·53
Nitrogen	..	..	1·63	2·06	2·77	3·36
Water vapour	..	..	·89	1·04	1·29	1.5

where the values given above multiplied by 10^{-6} = N, where N denotes the absolute viscosity in c.g.s. units. The conductivity increases with rise of temperature ; the value for air at 400° C. being double that at 20° C., whilst its viscosity is more than double.

These effects on heat transmission are opposite in direction ; thus the effect of temperature range (for the same drop) is, in practice, negligible.

It is generally known amongst engineers that the heat transfer between a gas and a metal is very small, but this fact is often overlooked in practice, and in some cases within the writer's knowledge, highly superheated steam has been supplied to heaters with the object of increasing the work done. An interesting case occurred recently, where two heaters were installed, the liquid passing through them in series ; each heater was connected to a supply of exhaust steam ; as a higher final temperature was required, live steam at 20—30 lb. to the sq. in. was supplied to one of the heaters, with the result that the final temperature dropped. Upon investigation it was found that the steam was superheated to a total temperature of 200° C.

Consider the case of a tubular heater supplied with saturated steam at 100° C. and heating water from 20° C. to 80° C., the steam passing through the tubes at the rate of 1 lb./sec./ft.^{2} sectional area (or W=1). The average temperature difference between the steam and water is 50° C. The overall coefficient, H, therefore would equal

$$\frac{1}{\dfrac{1}{10,700} + \dfrac{1}{6200}} = 3700/cals./m.^2/hour.$$

The effect of superheating the steam before entry to 200° C. would mean that the temperature difference would now be 118°.

The average absolute temperature of the steam (when giving up its superheat) is about 763° F., thus T = 763 and $\dfrac{Cp}{M^{0 \cdot 3}} = \dfrac{0 \cdot 48}{18^{0 \cdot 3}} = 0 \cdot 2$, and on reference to alinement chart (Fig. 6) it will be found that h=45.

Now the superheat and latent heat of the steam when entering is about 48+539, or 587 cals., thus the superheat is about 8·2% of the total available heat. The resistance to heat flow, therefore, is

$$\frac{3700}{45} \times \frac{50°}{118°}$$

or 34·8 times that of saturated steam. Since, however, there is only 8·2% of the total heat to be transmitted, the surface of the heater must be 34·8 × 8·2/100 = 2·85 times that required for the saturated steam for the same amount of heat transference. This rough calculation provides an illustration of the mistake of using superheated steam for heating or evaporation.

Very little data have been published upon the heat transfer between air and the metal wall of a tube. Carrier gives an equation

$$h = \frac{109W}{1 \cdot 42 + W}$$

for 1-in. diam. pipes which are arranged to give 50% of clear area, where W=mass velocity in lb. air flowing per second per sq. foot of cross-section of the heater or cooler, the air flowing at right angles to the tubes.

The curves of Fig. 8 show this equation ; it will be seen that any increase in the value of W above 20 has no appreciable affect upon the transmission coefficient.

It is well known that when the velocity of a fluid passing through a tube is low, the value of h is very small, but when the velocity increases and reaches a certain critical value which is dependent upon the viscosity and density of the fluid and upon the diameter of the pipe, the value of h increases very greatly.

In all heat transfer equations turbulent flow is assumed. On this point it may be well to mention that the rate of flow of water through a steel or iron pipe has a great effect upon the corrosion which takes place. Dr. Newton Friend in some recent experiments has shown

that when water flowed with a velocity lower than the critical, corrosion took place rapidly, but with turbulent flow the corrosion was much reduced.

A very ingenious experiment was carried out by M. Dieterlen at the Kestner laboratories in Paris in which a stream of water was directed on to the centre of a circular polished steel plate,

Carrier formula, $h = \dfrac{109\,W}{1\cdot42 + W}$ For air impinging on 1 in. diam. tubes, 50% of the space occupied by tubes.

FIG. 8.

ft. lb. per sq. ft. per sec. = 14.0 dynes/Cm²/sec.

$V = \dfrac{26.42\,n}{d'' \times Sp.\,gr.}$

Critical velocity of liquids flowing in circular pipes.
(O. Reynolds' criterion.)

FIG. 9.
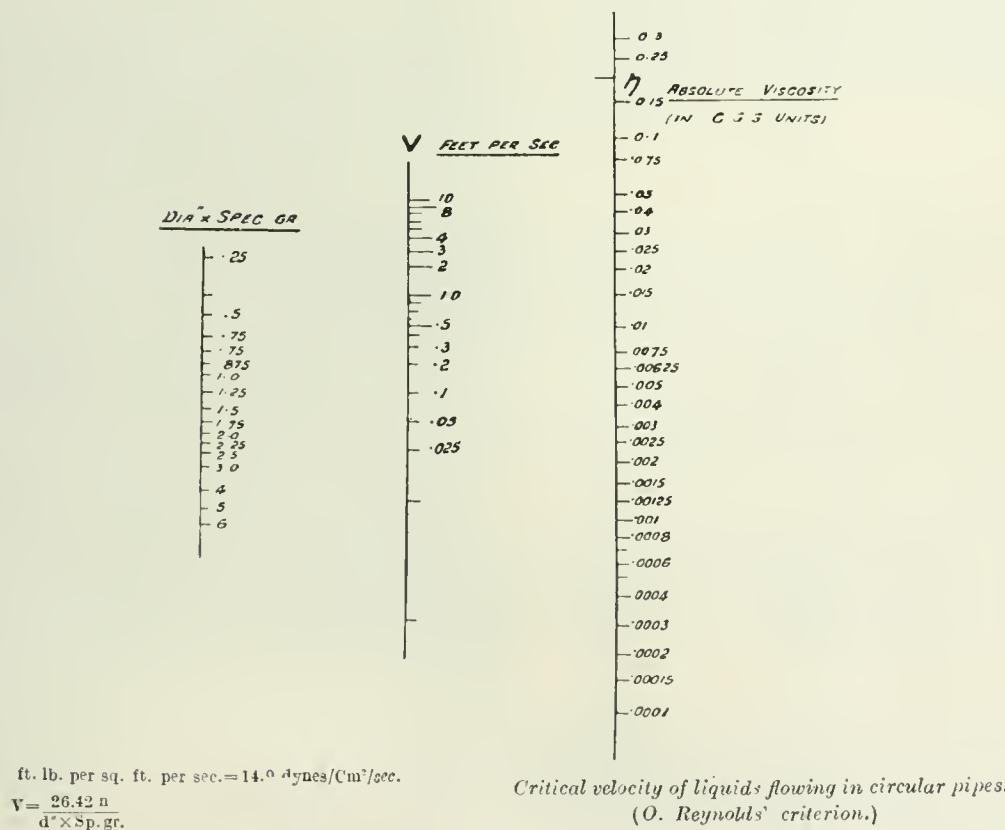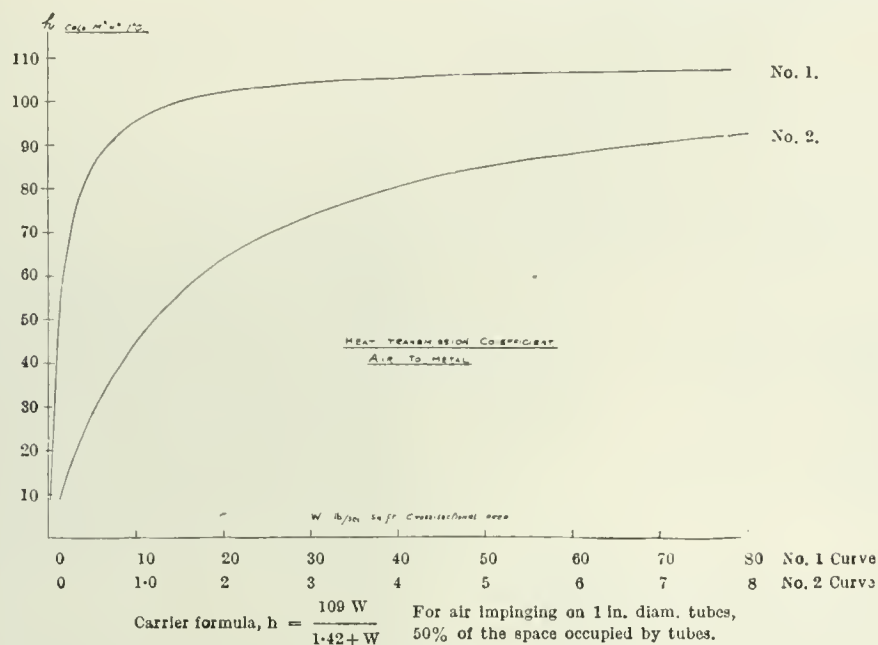
placed horizontally, the water flowing over the edge. The corrosion effects produced demonstrate the relative effects of turbulence and viscous flow, both of these regimes being obtained on the plate by regulating the flow. The central portion was hardly attacked, while the outer annulus was rapidly corroded. There is, therefore, every reason why in economisers and heat exchangers that turbulence should always be obtained. Alinement chart Fig. 9 will give approximate figures for critical velocity, but only applies to smooth pipes; those used commercially give a somewhat lower figure dependant on their roughness.
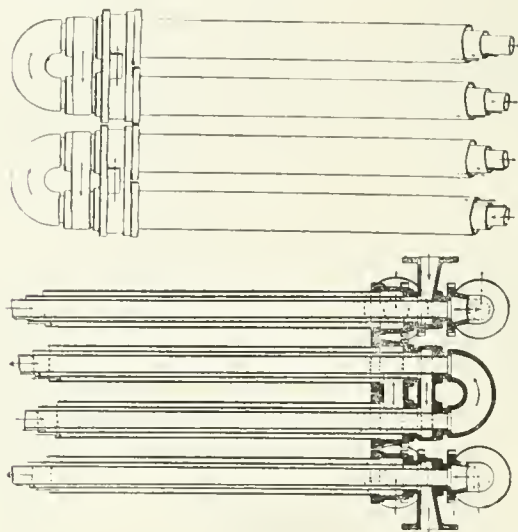
water flows in the middle annular passage, whilst vapour passes through the inner and outer tubes.

This concentric-tube type of heat exchanger allows of true counter-current flow. The liquids pass through the tubes and annular space in series, thus obtaining a relatively high velocity compared with the more usual multitubular type.

Fig. 11 shows another type of concentric tube heat-exchanger in which the whole of the tubes can be inspected and cleaned out by removing two end covers.

From the above considerations it will be seen that the concentric-tube type offers the optimum conditions for rapid heat transfer. This design also allows of the tubes being easily cleaned *in situ*, or where hard scale is deposited they can be removed without trouble.

This design has several valuable features. The high velocity reduces the tendency to scale being deposited on the interior surface of the tubes. Any scale formed on the central tube can usually be removed by passing a scraper or brush through the tubes. This operation is carried out very quickly in the exchanger shown of Fig. 10 by merely removing the end bends.

The large diameter of tube employed allows of a stronger scraper or brush and rod being used.

In concentric-tube heat exchangers, where the transfer is between water and a corrosive or scaling liquid, it must be so arranged that the corrosive liquid flows through the internal tube. The material of this tube must be

FIG. 10.

ELEVATION

END VIEW
with End Box Removed.

PLAN
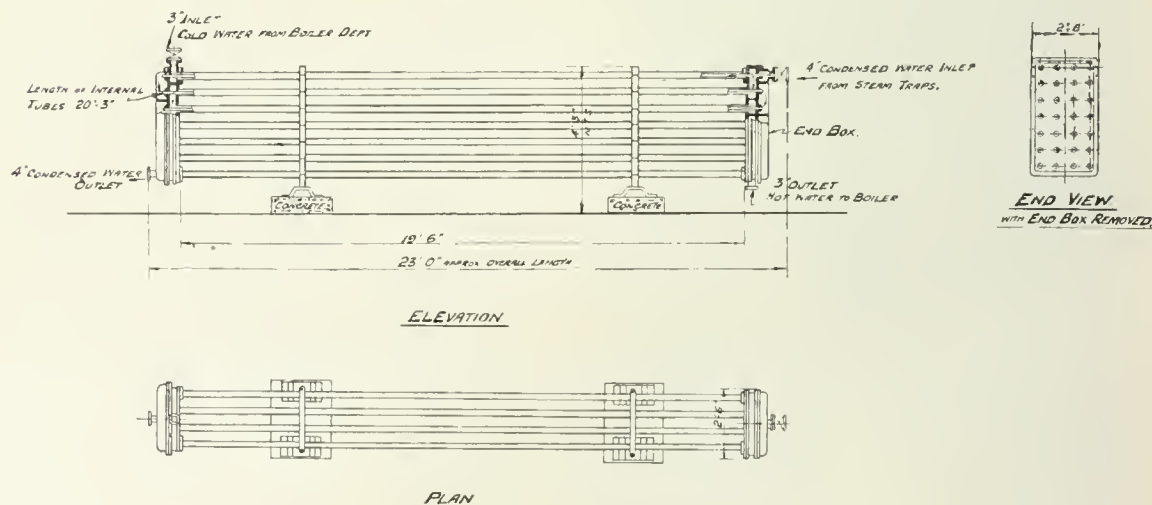
FIG. 11.

For rapid heating or cooling, one of the chief factors, therefore, is the velocity of the liquid.

Fig. 10 shows a type of heat exchanger which, whilst costly to manufacture, has an extremely high efficiency. The condensing

chosen with care to suit the nature of the liquid, and the chemical engineer is often faced with the difficult problem of obtaining a material which will be resistant to attack and also possess sufficient mechanical strength.

Unfortunately for us, nature has not often united in one material these two distinct properties; thus most of the so-called acid-resisting metals possess low mechanical strength. Again, cost often enters largely into the problem and in cases where a high degree of purity is not required in the liquid it is often more economical to use steel tubes and allow corrosion, if not too rapid, to take place, renewing the tubes at frequent intervals.

In contrast to this, when the slightest contamination of the liquid must not occur, silver and even golden tubes have been used for certain liquids. A frequent cause of corrosion on the water side of the tube is the presence of dissolved oxygen in the water due to its becoming aerated before entry.

The only remedy for this trouble is to de-gas the water by passing it through a special apparatus before entering the exchanger.

In order to reduce the corrosion in steel or iron tube heat exchangers, which takes place when they are not in operation, care should be taken that they are filled with distilled water. Experience has shown that this simple remedy is extremely effective in prolonging the life of the tubes.

Heat exchangers for flue gases.

In the design of economisers and air heaters a very important point arises. Where waste heat boilers or economisers are employed the water entering should be at a temperature slightly higher than the dew-point of the gases, as in most cases there is a large amount of moisture present, which otherwise would condense on the outer surface of the tubes, and in combination with sulphur dioxide would quickly set up very serious corrosion.

It has been shown already that the temperature rise across the water film is very small compared with that across the gas film; thus, the tube is at practically the same temperature as the water, the result being that the gas film in contact with the tube surface is also at approximately the same temperature, and thus any moisture will be deposited.

No such trouble is met where air heaters are used, as in that case the temperature difference across the air film on the inner surface of the tube is substantially equal to the temperature difference on the gas film; thus the temperature of the metal is approximately a mean between the gas and air temperature, and in most cases well above the dew-point of the gas. The above consideration shows the difficulty of satisfactorily utilising waste heat for heating up water initially at a low temperature.

Appendix.

A point to be considered in the design of heating coils and tubular heat exchangers is the determination of the correct ratio between the length of the coil, or total length of passage of the tube and its diameter. Let us consider the simple case of a coil through which a hot liquid is flowing immersed in a tank of a colder liquid, this latter liquid being kept at a constant temperature, T, and well agitated, the hot liquid cooling in its passage through the tube from t_1 to t_2.
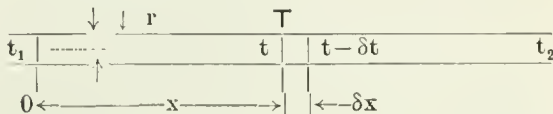
A pipe of inner radius, r (assuming the thickness of the pipe wall is negligible compared with the diameter) with an overall coefficient, H, carries a liquid of specific heat, c, density, d, velocity, v, entry temperature, t_1, temperature at a distance, x, along its length, t, and exit temperature after passing through length l of pipe, t_2. Temperature in tank, T, (kept uniform by agitation).

Then $\pi r^2 \, d \, c \, v \, \dfrac{dt}{dx} = -2\pi r \, H \, (t-T)$

or $\dfrac{1}{t-T} \dfrac{dt}{dx} = \dfrac{-2H}{r \, d \, c \, v}$

Integrating we have :

$\log_e (t-T) = \dfrac{-2Hx}{r \, d \, c \, v} + $ a constant,

when x=0, t=t_1,

$\therefore \log_e (t_1 - T) = $ the constant. When x=l, t=t_2,

$\therefore \log_e \dfrac{t-T}{t_1-T} = \dfrac{-2Hx}{r \, d \, c \, v}$, or $\dfrac{t-T}{t_1-T} = e^{\frac{-2Hx}{rdcv}}$

To find the total heat, Q, transferred through a pipe of length, l, we have :—

Total heat in unit time

$$= \int_0^l 2\pi r \, H \, (t-T) \, dx,$$

$$= \int_0^l 2\pi r \, H \, (t_1 - T) \, e^{\frac{-2Hx}{rdcv}} \, dx,$$

$$= \pi r^2 \, d \, c \, v \, (t_1 - T) \, (1 - e^{\frac{-2Hl}{rdcv}})$$

This equation shows that the heat transmitted is not proportional to l.

Let us consider how the total heat transmitted varies with the length. Writing Z for $\dfrac{-2Hl}{r \, d \, c \, v}$ the index of e, and Q for the total heat transmitted.

Then the value of Q for an infinite length of pipe would be given by the equation.

$Q = \pi r^2 \, d \, c \, v \, (t_1 - T)$ and half this value by

$$\pi r^2 \, d \, c \, v \, (t_1 - T) \left(1 - \dfrac{1}{e^z}\right)$$

when $\dfrac{1}{e^z}$=0·5 or Z=0·69.

Thus half the maximum amount of heat will be transmitted when :—

$\dfrac{2Hl}{r \, d \, c \, v}$=0·69 or $l = \dfrac{0·345 \, r \, d \, c \, v}{H}$

where all dimensions are metric units :—

r =internal radius of pipe in decimetres.

d =density in kg./decimetre³.

c =specific heat, water=1.

v =velocity in decimetres/hour.

H=overall coefficient of heat transmission in kg./cals. per square decimetre/hour.

l =length of pipe in decimetres.

Fig. 12 gives the percentage change of heat transmitted above or below half the maximum value for percentage changes in length as designated above.

Example.

Water at a temperature of 85° C. enters a coiled pipe placed in a tank of water which is maintained at a constant temperature of 15° C., by agitation.

Then :—

Choosing a velocity of 1·5 metres per second.

v =54,000 decimetres/hour.

T =15° C., t_1=85° C., t_2=25° C.

H=assumed overall coefficient of heat transmission, 15 major calories per decimetre²/hour 1° C.

Percentage increase over the length required to transmit half the maximum heat possible.

Percentage difference in tube length.
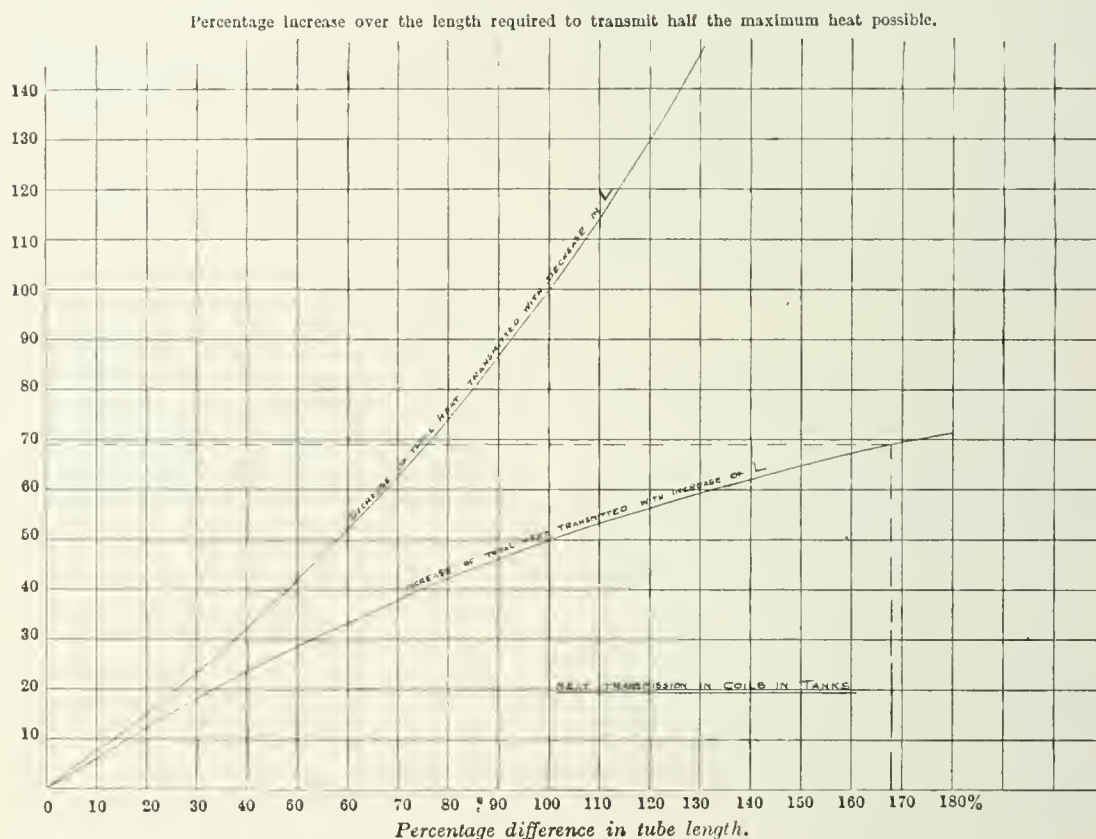
FIG. 12.

It will be found that l increases very rapidly as the value (t_2—T) decreases. The length l for a given temperature difference t_1—t_2 may be obtained as follows :—

$$Q=r^2 d c v (t_1-T) \left(1-\frac{l}{e^z}\right)$$

also

$$Q=r^2 d c v (t_1-t_2)$$

∴

$$e^z=\frac{t_1-T}{t_2-T}$$

or

$$Z = \log_e \frac{t_1-T}{t_2-T}=\frac{2H}{r d c v}$$

∴

$$l=\frac{r d c v}{2H} \log_e \left(\frac{t_1-T}{t_2-T}\right) \times \left(\frac{t_1-T}{t_2-T}\right)$$

Cross-sectional area of pipe$=\dfrac{15,000}{54,000}=0\cdot278$ dm.²

$$\gamma = \tfrac{1}{2}\sqrt{\frac{0\cdot278}{0\cdot7854}} =0\cdot298 \text{ dm. (inner radius of pipe).}$$

$$=\frac{r d c v}{2H} \log_e \frac{t_1-T}{t_2-T}$$

$$=\frac{0\cdot298\times 1\times 1\times 54,000}{2\times 15} \log_e \left(\frac{70}{10}\right)=1043 \text{ dm.}$$

Q (for half maximum value)

$=\pi. r^2. d, c. v. 0\cdot5 (t_1-T)$

$=\pi.0\cdot09, 54,000, 0\cdot5, 70=532,000$ cals.

equivalent value of $l = \dfrac{0.345 \ r \ d \ e \ v}{H}$

$$= \frac{0.345,\ 0.298,\ 54{,}000}{15} = 370 \text{ dm.}$$

This length of pipe would cool the liquid, 15 tons per hour,

$$\frac{532{,}000}{15{,}000} = 35.5° \text{ C.,} \ i.e.,\ \text{from } 85° \text{ C. to } 49.5° \text{ C.}$$

To cool down to 25° C. represents the transfer of 69% more heat. From the curve (Fig. 13) it will be found that to transmit this percentage increase in the heat the length l must be increased by 170%, or in other words l must be increased from 370 dm. to $370 \times 2.7 = 1010$ dm., which checks within slide rule accuracy with the value 1043 obtained by the equation given above.

The curve will be found useful for rapidly obtaining a suitable length of coil in cases where it is required to cool a liquid, but not to a pre-determined temperature, t_2.

It is also a very good illustration of the limitations of heating or cooling, by means of coils placed in a tank.

Heat exchange.

Case 1 (a). Steam coil in tank. The steam being condensed and the condensate leaving without being cooled.

Let T = steam temperature, constant.
  t_2 = initial temperature of the liquid in tank.
  t_1 = final temperature of the liquid in tank.
  t = time in hours to heat up the contents of the tank from t_2 to t_1.
  H = coefficient of heat transmission, 1° C.
  S = heating surface of coil.
  Q = (quantity of liquor in tank) × (its specific heat).

Let the rise of temperature in tank in time dt = dx. Then : HSdt (T—x) = Q dx = heat flowing in time dt, or $dt = \dfrac{Q}{HS} \cdot \dfrac{dx}{(T-x)}$

integrating, $t = \dfrac{Q}{HS} \dfrac{d\ x}{T-x} = \dfrac{Q}{HS} \log_e \dfrac{T-t_2}{T-t_1}$

which is similar to the well known equation for condensers and steam-heated heaters where

$$S = \frac{Q}{H} \log_e \frac{T-t_2}{T-t_1}$$

t_1 and t_2 being in this case the outlet and inlet temperatures of the liquid to be heated.

Case 2.—Heating up the contents of a tank by circulating the liquid through an external heater, which is heated by means of steam. This case may be sub-divided into :—

(a) Where the steam in the heater is at constant pressure.
(b) Where the steam in the heater is at constant temperature.

It will be seen that under condition (a) there is a great demand for steam on the boiler at the commencement of the heating as there will be then a maximum temperature difference and consequently a rapid heat flow.

In the second case there is no rush of steam at the commencement, but a partial vacuum may be formed in the steam space, rendering difficult the removal of the condensate, unless an extraction pump or barometric discharge is fitted.

(a) Constant steam pressure in the steam pipe and constant temperature difference in the heater.

Let Q = contents of tank in litres × specific heat of liquid.
  W = liquor circulated in litres per hour.
  S = heating surface of heater, in sq. metres.
  H = overall heat transmission coefficient expressed as calories/sq. metre/ 1° C./hour.
  X = time in hours.

It is assumed that the temperature in every part of the tank is the same.

Time in hours.
T Steam. t_2 leaving heater. t_1 entering heater.
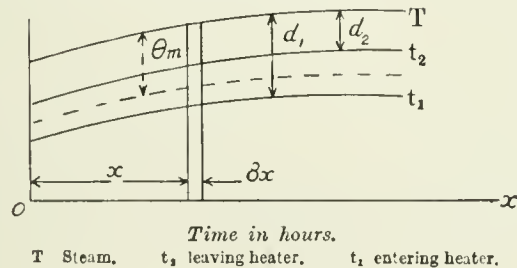
The mean temperature difference,

$$\theta_m = \frac{(T-t_1) - (T-t_2)}{\log_e \left(\dfrac{T-t_1}{T-t_2} \right)}$$

let us denote $(T-t_1)$ by d_1 and $(T-t_2)$ by d_2;

then

$$\theta_m = \frac{d_1 - d_2}{\log_e \dfrac{d_1}{d_2}}$$

The heat transmitted to the heater during the time dx is $S.H.\theta_m.dx$ calories. This heat raises the temperature of the liquid $d_1 - d_2$ degrees in passing through the heater,

or $S.H.\theta_m.dx = W.(d_1 - d_2) dx$

substituting values of θ_m in terms of d_1 and d_2

$$S\ H\ \frac{d_1 - d_2}{\log_e \left(\dfrac{d_1}{d_2} \right)} = W\ (d_1 - d_2)$$

or $\dfrac{S\ H}{W} = \log_e \dfrac{d_1}{d_2}$ hence $e^{\frac{SH}{W}} = \dfrac{d_1}{d_2}$

Thus $\dfrac{d_1}{d_2}$ can be evaluated for a given case, it being assumed that the quantities S H and W are constant.

The time x required to raise the contents of the tank to a given temperature may be found as follows.

The heat transmitted to the liquid in time dx is $W(t_2 - t_1)\,dx_1$; this heat causes a rise in temperature of the tank contents of dt.
or $W(t_2 - t_1)\,dx = Q\,dt$.

Now as the temperature difference θ_m is constant $\dfrac{dx}{dt} = \dfrac{Q}{W}\,\dfrac{1}{t_2 - t_1}$ is constant.

Thus $x = \dfrac{Q}{W}\,\dfrac{t_1}{(d_1 - d_2)}$ where t_1 is the total temperature rise of Q.

The steam consumption over a given period is obtained as follows :—

At a given time, x, the heat transmitted to the liquid is at the rate of $W(t_2 - t_1)$ calories per hour.

Let R = the steam consumption over a given period x,

H_1 = total heat of steam at temperature T.

Then $\dfrac{dR}{dx} = \dfrac{W}{H_1 - T}\,(t_2 - t_1)$ calories .. (a)

now $\dfrac{dx}{dt_1} = \dfrac{Q}{W}\,\dfrac{1}{t_2 - t_1}$

$\therefore \quad (t_2 - t_1) = \dfrac{Q}{W}\,\dfrac{dt}{dx}$

thus $\dfrac{dR}{dx} = \dfrac{Q\,\frac{dt}{dx}}{H - T}$

also $\dfrac{dt_1}{dx} = \dfrac{W}{Q}\,(d_1 - d_2)$

Integrating

$$t_1 = \frac{W}{Q}(d_1 - d_2)\,x + t_2$$

where t is the temperature in the tank before heating,
again $T = t_1 + d_1$

$\therefore \quad T = \dfrac{W}{Q}(d_1 - d_2)\,x + t + d_1$

subst. in (a).

$$\frac{dR}{dx} = \frac{W(d_1 - d_2)}{H - \frac{W}{Q}(d_1 - d_2)\,x - t - d_1}$$

integrating

$$R = Q_1 \log_e\left(\frac{H - d_1 - t}{H - d_1 - t - \frac{W}{Q}(d_1 - d_2)\,x}\right)$$

The steam consumption at constant pressure, and temperature, T, may be determined as follows :—

$$\frac{dR}{dx} = \frac{Q\,\frac{dt_1}{dx}}{H - T}$$

$\therefore \quad dR = \dfrac{Q}{H - T}\,dt \qquad \therefore \quad R = \dfrac{Q}{H - T}\,(t_2 - t_1)$

To find the time required to heat up the liquid in the tank to a given temperature, t_b

$$\frac{dt_1}{dx} = \frac{W}{Q}(t_2 - t_1) = \frac{W}{Q}(d_1 - d_2)$$

now $\quad d_2 = \dfrac{d_1}{e^{\frac{SH}{W}}}$

$\therefore \quad \dfrac{dt_1}{dx} = \dfrac{W}{Q}\,d_1\,\dfrac{e^{\frac{SH}{W}} - 1}{e^{\frac{SH}{W}}}$

now $\quad d_1 = T - t_1$

$\therefore \quad \dfrac{dt_1}{dx} = \dfrac{W}{Q}\,\dfrac{e^{\frac{SH}{W}} - 1}{e^{\frac{SH}{W}}}\,(T - t_1)$

and $\quad \dfrac{dt_1}{T - t_1} = \dfrac{W}{Q}\,\dfrac{e^{\frac{SH}{W}} - 1}{e^{\frac{SH}{W}}}\,dx$

Integrating, between the limits t_a, t_b

$$x = \frac{Q}{W}\,\frac{e^{\frac{SH}{W}}}{e^{\frac{SH}{W}} - 1}\,\log_e\left(\frac{T - t_a}{T - t_b}\right) \qquad .. \quad (1)$$

It may be interesting to compare the two methods of heating applied to a concrete case.

A tank contains 15,000 litres of water at 15° C. steam at a pressure of 60 lb. inch² (gauge). T=153° C. Heater surface S= 16 m². H=500 (assumed). Water circulated, 24,000 litres hour.

It is required to bring the contents of the tank up to boiling point.

(a) By constant temperature difference in the heater values :—

$$T = 153 \qquad H_1 = 653 \qquad Q = 15{,}000$$
$$H = 1500 \qquad S = 16 \qquad W = 24{,}000$$

now $\quad \dfrac{SH}{W} = \log_e \dfrac{d_1}{d_2} \qquad \therefore \quad e^{\frac{SH}{W}} = \dfrac{d_1}{d_2}$

but $\quad \dfrac{SH}{W} = 1 \qquad$ thus $\dfrac{d_1}{d_2} = e$

Assuming $d_1 = 53°$ C., as the final temperature in the tank is to be 100° C.,

then $\quad \dfrac{53}{e} = 19 \cdot 5 = d_2.$

To find the time required to heat up the tank contents to 100° C., i.e., 85° rise in temperature :

$$\frac{dx}{dt} = \frac{Q}{W}\,\frac{1}{d_1 - d_2} \text{ (a constant)}$$

$\therefore \quad x = \dfrac{15000 \times 85}{24000 \times 33 \cdot 5} = 1 \cdot 585$ hours.

The steam consumption will be :

$$R = 15000 \log_e\left(\frac{653 - 53 - 15}{653 - 53 - 15 - \frac{24{,}000}{15{,}000}(53 - 19 \cdot 5)1 \cdot 585}\right)$$

or R = 2350 kg.

An alternative and simpler method, which may be used as a check, is as follows :—

The initial steam temperature is 68° C. (see diagram).
The final steam temperature is 153° C.
Thus the mean temperature is 110·5° C.

The mean latent heat is $653 - 110 \cdot 5 = 542 \cdot 5$ cals./kg.

The heat required to raise the tank contents from $15°$ C. to $100°$ C. is $15,000 \times 85 = 1,275,000$ calories.

Thus the steam required is

$$R = \frac{1275000}{542 \cdot 5} = 2350$$

(b) By constant steam pressure in the heater. To find the time required to heat up the tank contents, substituting values in (1)

$$x = \frac{15000}{24000} \cdot \frac{e^{\frac{SH}{W}}}{e^{\frac{SH}{W}} - 1} \log_e \frac{153 - 15}{153 - 100}$$

$$= \frac{15000}{24000} \cdot \frac{2 \cdot 718}{1 \cdot 718} \times 0 \cdot 96 = 1 \cdot 03 \text{ hours.}$$

The steam consumption will be

$$R = \frac{15000}{500} \times 85 = 2550 \text{ kg.}$$

Comparing the figures obtained above for two methods of heating it is seen that with constant temperature difference the steam consumption is about 8% lower than when working with constant pressure in the heater, but that the rate of heating is very much slower. This is more marked where higher steam pressures are employed. The designer must be guided in his choice of methods by such considerations as the capacity of the boiler, time of heating, and capital cost. This method of heating the contents of tanks is much superior to the employment of steam coils, as a relatively small heating surface is required, due to the high velocity at which the liquid passes through the tubes. The ease with which the tubes can be cleaned, the heaters being usually of the tubular type, is to be noted, and the agitation produced in the tank, which is generally an advantage in chemical process work.

In order to facilitate the calculation of the cooling or heating surface in tubular heat-exchangers some equations and tables, due to Gröber, are given

Logarithmic mean temperature difference

(a) *Parallel flow.*
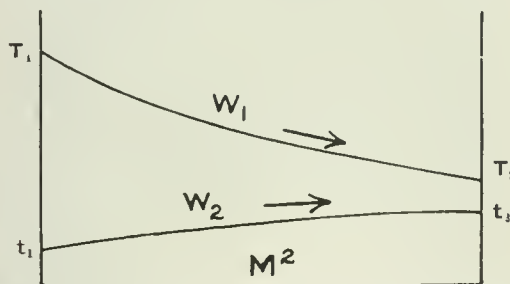
W_1 and $W_2 =$ (Litres flowing per hour) $\times$ (Specific heat) $\times$ (Specific gravity) of the hotter and colder liquid respectively.

H   = Co-efficient of heat transmission, Cals./M²/hr./1° C.

M² = Heating or cooling surface in square metres.

Q   = Total heat transferred, in calories/hour.

The final temperatures T_2 and t_2 for a given surface can be obtained as follows, T_1 and t_1 being initial temperatures :—

$$Q = (T_1 - t_1) W_1 \quad \text{Function of} \left(\frac{W_1}{W_2} \quad \frac{HM^2}{W_1} \right)$$

Table I. gives values of this function for different values of $\frac{W_1}{W_2}$ and $\frac{HM^2}{W_1}$ thus Q can be evaluated for a given case, and $T_1 - T_2 = \frac{Q}{W_1} \therefore T_2$ is known.

In the same way $t_1 - t_2 = \frac{Q}{W_2} \therefore t_2$ is known.

Example.—In a parallel current cooler hot liquid of specific heat $0 \cdot 727$ and specific gravity $1 \cdot 1$, flows at the rate of 250 litres per hour, entering at a temperature of $120°$ C. It is cooled by water entering at $10°$ C. and flowing at the rate of 1000 litres hour.

We have :—
$W_1 = 250 \times 0 \cdot 727 \times 1 \cdot 1 = 200$
$W_2 = 1000 \times 1 \cdot 0 \times 1 \cdot 0 = 1000$
$\frac{W_1}{W_2} = \frac{200}{1000} = \frac{1}{5}$
$\frac{HM^2}{W_1} = \frac{20 \times 5}{200} = \frac{1}{2}$

From Table I. Function $\left(\frac{1}{5}, \frac{1}{2} \right) = 0 \cdot 38$

$W_1 (T_1 - T_2) = 200 (120 - 10) = 22,000.$
$\therefore Q = 22,000 \times 0 \cdot 38 = 8360$ calories/hour.

$T_1 - T_2 = \frac{8360}{200} = 41 \cdot 8°$ C., or $T_2 = 78 \cdot 2°$ C.

$t_1 - t_2 = \frac{8360}{1000} = 8 \cdot 36°$ C, or $t_2 = 18 \cdot 36°$ C.

(b) *Counter flow.*

In a like manner the final temperatures can be determined, using the value of the function of $\left(\frac{W_1}{W_2} \quad \frac{HM^2}{W_1} \right)$ obtained from Table II.

Comparison between parallel and counter current flow :—

Fig. (a)
Parallel flow ($W_1 < W_2$)

Fig. (b)
Counter flow ($W_1 < W_2$)

Fig. (c)
Counter flow ($W_1 > W_2$)

Denote the temperature difference at the inlet of the warmer fluid by θ_1 and at the outlet by θ_2. Then

$$\left(\frac{\theta_1}{\theta_2}\right) = e - \left(\frac{1}{W_1} \pm \frac{1}{W_2}\right) HM^2 \quad \text{(Gröber)}$$

in which the negative sign is taken for counter current and the positive sign for parallel flow.

Denoting the exponent $\left(\frac{1}{W_1} \pm \frac{1}{W_2}\right) HM^2$ by x, if θ_2 is smaller than θ_1, x is negative, if larger x is positive.

Thus, for parallel flow

$$x = -\left(\frac{1}{W_1} + \frac{1}{W_2}\right) HM^2.$$

In counter flow

$$x = -\left(\frac{1}{W_1} - \frac{1}{W_2}\right) HM^2.$$

(For values of e^x and e^{-x} see Table IV.).

Firstly.—When W_2 is greater than W_1, θ_2 is smaller than θ_1 and x is negative. See Fig. (b).

Secondly.—When W_1 is greater than W_2, θ_2 is greater than θ_1 and x is positive. See Fig. (c).

To determine the logarithmic mean temperature difference of θ_m:—

$$\theta_m = \theta_1 \text{ function of } \left(\frac{\theta_2}{\theta_1}\right)$$

From Table V. values of $\frac{\theta_m}{\theta_1}$ and functions of $\frac{\theta_2}{\theta_1}$ are given.

Example,

$$\theta_1 = 40° \text{ C.} \qquad \theta_2 = 80° \text{ C.}$$

$$\frac{\theta_2}{\theta_1} = \frac{80}{40} = 2$$

and function $\left(\frac{\theta_m}{\theta_1}\right) = 1\cdot44$ (from Table V.).

$$\therefore \quad \theta_m = 40 \times 1\cdot44 = 57\cdot6° \text{ C.}$$

Determination of the necessary heating or cooling surface.

Example   Parallel flow.
$W_1 = 20$ $T_1 = 120°$ C.
$H = 20$ $T_2 = 58°$ C.
$W_2 = 400$ $t_1 = 20$
$Q = 20 (120 - 58) = 20 \times 62.$

$$\text{Function} \left(\frac{W_1}{W_2} \frac{HM^2}{W_1}\right) = \frac{Q}{W_1(T_1 - t_1)} = \frac{20 \times 62}{20(120-20)}$$
$$= 0\cdot62$$

From Table I. we find from

$$\frac{W_1}{W_2} = \frac{1}{20} \text{ and function value } 0\cdot62$$

$$\text{that } \frac{HM^2}{W_1} = 1 \qquad \therefore M^2 = 2$$

TABLE I.
Heat exchange with parallel flow.

$\frac{HM^2}{W_1}$	$\frac{1}{30}$	$\frac{1}{10}$	$\frac{1}{3}$	$\frac{1}{2}$	1	2	3	∞
$\frac{W_1}{W_2} = 0$	0·033	0·10	0·28	0·39	0·63	0·86	0·96	1·00
$\frac{1}{100}$	0·033	0·10	0·28	0·39	0·63	0·86	0·95	0·99
$\frac{1}{20}$	0·033	0·10	0·28	0·39	0·62	0·84	0·91	0·95
$\frac{1}{10}$	0·033	0·10	0·28	0·38	0·61	0·81	0·89	0·91
$\frac{1}{5}$	0·033	0·10	0·27	0·38	0·58	0·76	0·81	0·83
$\frac{1}{2}$	0·033	0·10	0·26	0·35	0·52	0·63	0·66	0·67
1	0·033	0·09	0·25	0·32	0·43	0·49	0·50	0·50
2	0·033	0·09	0·21	0·26	0·32	0·33	0·33	0·33
5	0·032	0·08	0·14	0·16	0·17	0·17	0·17	0·17
10	0·028	0·06	0·09	0·09	0·09	0·09	0·09	0·09
20	0·024	0·04	0·05	0·05	0·05	0·05	0·05	0·05
50	0·016	0·02	0·02	0·02	0·02	0·02	0·02	0·02
100	0·009	0·01	0·01	0·01	0·01	0·01	0·01	0·01
∞	0·000	0·00	0·00	0·00	0·00	0·00	0·00	0·00

TABLE II.
Heat exchange with counterflow.

$\frac{HM^2}{W_1}$	$\frac{1}{30}$	$\frac{1}{10}$	$\frac{1}{3}$	$\frac{1}{2}$	1	2	3	00
$\frac{W_1}{W_2} = 0$	0·033	0·10	0·28	0·39	0·63	0·86	0·95	1·00
$\frac{1}{100}$	0·033	0·10	0·28	0·39	0·63	0·86	0·95	1·00
$\frac{1}{20}$	0·033	0·10	0·28	0·39	0·62	0·86	0·94	1·00
$\frac{1}{10}$	0·033	0·10	0·28	0·38	0·61	0·85	0·94	1·00
$\frac{1}{5}$	0·033	0·10	0·28	0·38	0·60	0·83	0·93	1·00
$\frac{1}{2}$	0·033	0·10	0·26	0·36	0·57	0·78	0·89	1·00
1	0·033	0·10	0·25	0·34	0·51	0·68	0·77	1·00
2	0·033	0·09	0·23	0·29	0·39	0·46	0·49	0·50
5	0·032	0·08	0·16	0·18	0·20	0·20	0·20	0·20
10	0·028	0·06	0·10	0·10	0·10	0·10	0·10	0·10
20	0·024	0·04	0·05	0·05	0·05	0·05	0·05	0·05
50	0·016	0·02	0·02	0·02	0·02	0·02	0·02	0·02
100	0·010	0·01	0·01	0·01	0·01	0·01	0·01	0·01
00	0·000	0·00	0·00	0·00	0·00	0·00	0·00	0·00

TABLE III.

Ratio $\dfrac{\text{Parallel flow}}{\text{Counter flow}}$

$\dfrac{HM^2}{W_1}$	$\dfrac{1}{10}$	$\dfrac{1}{3}$	$\dfrac{1}{2}$	1	2	3	∞
$\dfrac{W_1}{W_2}=0$	1·00	1·00	1·00	1·00	1·00	1·00	1·00
$\dfrac{1}{20}$	1·00	1·00	1·00	1·00	0·98	0·97	0·95
$\dfrac{1}{5}$	1·00	1·00	1·00	0·97	0·92	0·87	0·83
1	1·00	1·00	0·94	0·84	0·72	0·65	0·50
5	1·00	0·88	0·89	0·85	0·85	0·85	0·85
20	1·00	1·00	1·00	1·00	1·00	1·00	1·00
∞	1·00	1·00	1·00	1·00	1·00	1·00	1·00

Nature of gas.	$N, \times 10^6$	C
Oxygen	1·84	128
Air	1·69	114
Nitrogen	1·63	110
Carbonic acid gas	1·40	260
Water vapour	0·89	0
Hydrogen	0·85	74

TABLE IV.

(Exponential function).
e^{+x} and e^{-x}.

x	e^{+x}	e^{-x}	x	e^{+x}	e^{-x}	x	e^{+x}	e^{-x}
0·0	1·00	1·00	1·5	4·50	0·22	3·0	20·1	0·050
0·1	1·11	0·90	1·6	4·95	0·20	3·1	22·0	0·045
0·2	1·22	0·82	1·7	5·45	0·18	3·2	24·5	0·041
0·3	1·34	0·74	1·8	6·05	0·17	3·3	27·0	0·037
0·4	1·49	0·67	1·9	6·63	0·15	3·4	30·0	0·033
0·5	1·67	0·61	2·0	7·39	0·14	3·5	33·1	0·030
0·6	1·82	0·55	2·1	8·12	0·12	3·6	36·6	0·027
0·7	2·00	0·49	2·2	9·03	0·11	3·7	40·5	0·025
0·8	2·22	0·45	2·3	9·98	0·10	3·8	44·7	0·022
0·9	2·48	0·41	2·4	11·00	0·091	3·9	49·2	0·020
1·0	2·72	0·37	2·5	12·30	0·083	4·0	54·6	0·018
1·1	3·00	0·33	2·6	13·50	0·074	4·1	59·9	0·017
1·2	3·32	0·30	2·7	14·80	0·067	4·2	66·7	0·015
1·3	3·60	0·27	2·8	16·40	0·061	4·3	74·0	0·014
1·4	4·06	0·25	2·9	18·20	0·055	4·4	81·5	0·012

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TABLE V.

(Logarithmic mean temperature difference.)

$\dfrac{\theta_2}{\theta_1}$	$\dfrac{\theta_m}{\theta_1}$	$\dfrac{\theta_2}{\theta_1}$	$\dfrac{\theta_m}{\theta_1}$	$\dfrac{\theta_2}{\theta_1}$	$\dfrac{\theta_m}{\theta_1}$	$\dfrac{\theta_2}{\theta_1}$	$\dfrac{\theta_m}{\theta_1}$
0·05	0·32	0·18	0·48	1·00	1·00	5·0	2·49
0·06	0·34	0·19	0·49	1·10	1·05	6·0	2·79
0·07	0·35	0·20	0·50	1·20	1·10	7·0	3·09
0·08	0·37	0·25	0·54	1·30	1·14	8·0	3·36
0·09	0·38	0·30	0·58	1·40	1·19	9·0	3·64
0·10	0·39	0·35	0·62	1·50	1·23	10·0	3·91
0·11	0·41	0·40	0·66	1·60	1·28	11·0	4·17
0·12	0·42	0·45	0·69	1·70	1·32	12·0	4·43
0·13	0·43	0·50	0·72	1·80	1·36	13·0	4·68
0·14	0·44	0·60	0·79	1·90	1·40	14·0	4·93
0·15	0·45	0·70	0·84	2·00	1·44	15·0	5·17
0·16	0·46	0·80	0·89	3·00	1·82	16·0	5·41
0·17	0·47	0·90	0·95	4·00	2·16	20·0	6·34

Absolute viscosity N of gases and vapours

Value of $N \times 10^6$
For example air at 300 C : $N = 0.000\ 00289$ Kg./sec. M².

Nature of gas.	0° C.	100° C.	200° C.	300° C.	400° C.	500° C.
Oxygen	1·84	2·34	2·79	3·19	3·56	3·89
Air	1·69	2·15	2·53	2·89	3·21	3·53
Nitrogen	1·63	2·06	2·44	2·77	3·08	3·36
CO_2	1·40	1·88	2·32	2·72	3·09	3·43
Water vapour	0·89	1·04	1·17	1·29	1·39	1·50
Hydrogen	0·85	1·05	1·23	1·38	1·53	1·66

This table is calculated by means of the following formula:—

$$N_T = N_0 \frac{1 + \dfrac{C}{273}}{1 + \dfrac{C}{T}} \sqrt{\frac{T}{273}}$$ from the following values.

DISCUSSION.

Prof. J. W. HINCHLEY said the subject treated was made more difficult than was necessary through the non-adoption of reciprocals where simplification occurred. For instance, the resistivity of a material was a much more simple thing to deal with in chemical engineering problems than was conductivity. It was not necessary then to have $\dfrac{1}{h_1} + \dfrac{1}{h_2}$. By multiplying by the thickness one could get the resistance and get a simple Ohm's law in heat straightaway, in which the number of calories transmitted through a given amount of material is proportional to the area, multiplied by the temperature difference and divided by the resistance. The C.G.S. units were very excellent, but it was easy to have multipliers by which more satisfactory units could be used. A cubic unit could be taken as the unit rather than the centimetre, and the hour as the unit of time. In that way it was possible to get resistivity figures which ran from about 360 for copper downwards. It was certainly better to get simple figures. Having said this much about units, he hoped we should in future use metric units right through. It might be the excuse that other units made it easy to check, but he could assure them that there were far more blunders made by mixing up units, as the author had done, than people realised, and it was much better to keep to accurate calculations for such work as film resistances, etc., in metric units, and use rough figures in English units. The film conception of dealing with these problems, of course, was only a convenient device for dealing with a difficult problem. It seemed to him that there was turbulent flow, possibly in the centre of the tube, and viscous flow on the film. Therefore, there was not a stationary film and the concept of film was not absolutely true. It was, however, convenient like other chemical engineering devices, and the idea of flow coefficient could not be done away with and would always be very valuable in practice. One could study the fill con-

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ception before building the plant and derive useful results in that way. The author did not make any reference to the work of Fessenden in America who developed for the transmission of hot gases through pipes the log formula which seemed to give more accurate results than any other formula he knew. It was rather complicated, but it did give remarkably accurate results for the transmission of heat from gases to liquids. It was, of course, impossible for the author to mention all the difficulties met with in dealing with these problems. There was, for instance, the fact that when one put a thermometer in a hot space, the temperature was not right within any reasonable degree of error. In ordinary circumstances the temperature which a thermometer gave in spaces in which the vessel was at a different temperature from the actual air and heat transmission was taking place was very often considerably wrong, and he personally felt that a large number of experiments that had been made on this subject required correction through that error. The author had mentioned latent heat. It had to be remembered in this connexion that the conduction of heat from steam to any other liquid through a metal wall was different from the conduction in the case of benzene. The latent heat of benzene was 90, but the latent heat of water was 540, and from this one could realise that as soon as transmission of heat began, condensation began at the same time with saturated steam, and the whole of that latent heat was liberated so that we were not dealing with the same conditions as when dealing with a permanent gas. Latent heat must have a considerable influence in the total heat transmission, and it seemed to him that the latent heat had quite as much to do with the actual increase of temperature of the metals dividing the surface as the viscosity or the dynamic viscosity of the liquid film. He was immensely interested in the large amount of work the author had placed before them, but he could not help saying that they should be very careful of alignment charts. They were very nice things but he had seen so many of them which were inaccurate that he was always afraid of them. If they plotted a curve, that told a story, but the alignment chart never told them when they were wrong. If the man had put the rule in the wrong place, they never knew, and he therefore suggested that they should view with extreme suspicion the alignment chart.

Mr. P. PARRISH said they ought to devise means to overcome this question of the film, and indeed, if they looked at the ordinary practical applications they would find that this question of the film had to a very large extent already been dealt with. If they took the Bonecourt boiler at the outset, eight or nine years ago, that was then a very inefficient type of instrument, due to the fact that there was a straight line flow with very little turbulence, and practically no transmission of heat through the tubes, but by the adoption of a type of filler that difficulty of the straight line flow had been overcome to a very large extent, and the rate of transmission had been increased very materially. In dealing with these formulæ it was necessary to take into consideration the packing mediums which had been devised in a number of apparatus, such as those the author had been dealing with. Then he would like to refer to another type of apparatus, namely, the ordinary condenser in connexion with coke oven plant. He knew of plants in the northern portion of England where there were two distinct units. One was where the tubes and the gas travelled parallel to each other, and in the other case there were horizontal tubes, and the gases were passing at right angles to the tubes. The difference in the amount of cooling surface in the two cases was extraordinary. In one works at Middlesbrough there was a very striking example of this. On the one hand there were these enormous coolers, in which the gases travelled parallel to the tubes, as compared with the one in which the tubes were horizontal and the gases travelled at right angles to them, with the result that turbulence was set up.

Mr. N. SWINDIN said that, so far as he knew, the earliest paper on the subject was by Reynolds in 1874, and then velocity was made to play the major part in the flow through a pipe. Since then there had been a paper by Stanton in 1884, which was practically forgotten, but in it was given a complete law for transmission on each side of a plate. This paper took into account that K was a function of viscosity. It took into account the temperature of the pipe, the temperature of the wall, the temperature of the liquid against the wall, and the variation of viscosity with temperature, and it gave $K = 0.007$ and $K = .01$. He looked into this and found a straight line flow on one side and a turbulent flow on the other side of the plate, and the result was that he had arrived at two simple formulæ, one for straight line flow and the other for turbulent flow, which were as follows :—

For stream line flow $K = 250 v^{.5}$
where $K = $ Kg. cals./m², hr/°C.
   $K = $ Coefficient.
   $v = $ m.ps.vel.
 For turbulent flow—
$K = 90(1 + 0.004 T_0)(1 + 0.01t)v^{.84} p^{0.16}$
where T_0 is temperature of plate
   t is temperature of liquid in contact with plate.

The question of getting the temperature of the film was a very difficult one and there were practically no instruments which had been devised for the purpose. He had been interested

in obtaining the coefficient of K at temperatures about freezing point, and after experimenting at low temperatures he was able to verify Reynolds' criterion as the index for the influence of viscosity on heat flow.

The effect of a film of air or water was so enormous that it had led to the idea of covering metals with rubber to develop a heater for acid liquors. The conductivity of a metal was practically anything within 295 and 289 and no metal he knew would stand hydrochloric acid for any length of time, but if a film of rubber was put on to the outside of the metal, a transmission was obtained of practically one-fifth of a copper plate. Another point was that more heat could be got through a very thick wall tube than a very thin wall tube. He had been experimenting with $3\frac{1}{2}$ in. between the heat and the water, but more heat would go through that thickness than through 1-16th inch plate, due to the fact that the presence of the water on the other side cooled the tube to such an extent that it increased the viscosity of the film through which the heat had to pass. Therefore, in addition to increasing the velocity of the gas through the tube, it is a very wise thing to delay the heat passing through the tube. To give some idea of the small figures obtained in practice for K, it was mentioned that in the case of a B and W boiler it is about 60 ; with a Lancashire boiler it is about 20 ; in the case of a cast-iron vessel with $\frac{1}{8}$ in. enamel put into a jacket 3-16th in. with a space of $\frac{5}{8}$ in. between, K was about 110. Thus in the latter case, with two thicknesses of enamel, two thicknesses of metal and $\frac{5}{8}$ in. lead, more heat was transmitted than through the tubes of a B and W boiler.

The CHAIRMAN said there was one point not touched upon, and yet was probably of more importance in regard to this question than anything else, namely, the air or non-condensable vapours. The majority of heating was done with steam, and there was a certain amount of air in the vapour, but so far as he knew there had been no work done on the question as to what part of the vessel they should look to for the air. He knew of no definite work which had been done on this subject here, although there had been a considerable amount of research put into it on the Continent, and one of his friends who had been working on the subject had told him that, so far as he could see, where there was non-

condensable vapour or air in the tubular surface inside the shell there were curious patches where no work was being done on the surface simply because of the effect of this cloud of non-condensable vapour passing over the surface. We were still a long way from making theory come near to actual practice in many of these heat problems, and he believed that, as far as steam questions were concerned, if we could get to the bottom of this air problem by being able to free the steam from vapours or non-condensable gases, we should get practice much nearer to theory.

Mr. HEASTIE, replying to the discussion, referred first to Prof. Hinchley's criticism of alignment charts. He had, he said, been interested in alignment charts for a great many purposes besides heat transmission, and when dealing with a new investigation where the formulæ was as complicated as some he had shown, the only way to do it was to construct an alignment chart. He had found that the alignment chart was a very good servant, but a very bad master, and it was necessary to be very careful on that point.

Mr. Parrish raised the point about the Bone-court boiler and heat transmission and said the improvement was due to the turbulent flow. Personally, he (the author) was under the impression that a certain amount of it was due to the radiant heat in the tubes.

In reply to the Chairman, he had had a great deal to do with non-condensable vapours in evaporators. A case in point was a triple effect evaporator in which the steam was 20 lb. in the first effect, and the temperature measured on the thermometer was equivalent to 20 lb. saturated steam. On the second effect it was also 20 lb., but it was possible to put one's hand on the calandria, whilst the third effect was practically down to vacuum. It was found that this was all due to a huge air lock in the shell. The air pressure was simply due to the air accumulated, and it was a most remarkable thing to see the high pressure and correspondingly low temperature. In the design of these coolers the first thing the designer should bear in mind was to get away the vapour and air as quickly as possible, because if any air was present all coefficients and alignment charts went by the board.

A hearty vote of thanks was accorded the author.

THE EXTRACTION OF OIL FROM SEEDS, NUTS AND KERNELS.

By J. W. BREWIS.

There are few mills in this country in a position to concentrate on the extraction of oil from one or two particular seeds. Fluctuations in the prices of seeds, oils, cakes, the frequent small margin of profit after crushing and the fact that the demand for certain cakes is seasonal make such a procedure impossible for the majority of mills.

It may be necessary to extract the oil from Cotton, Til or Rape seeds, Soya Beans, Ground-nuts, Babassu Kernels, Copra, Palm Kernels, Shea nuts, Mowha seed, Linseed or Castor seed, and as practically each of these seeds and nuts possesses some distinct peculiarity, calling for special treatment, few mills are in a position to handle the complete range in the most efficient manner. The method of extracting the oil is frequently a matter of compromise—to suit the available plant. The production of crude edible oil from certain seeds with limited plant is a familiar difficulty, yet some surprising results are obtained, for instance, from open type presses, which were a few years ago considered unsuitable for rich oil seeds. It is, however, impossible to recommend a definite type of plant to handle each seed with equal over-all efficiency, and the problem of obtaining the highest efficiency on a wide range of seeds on a particular plant is likely to remain—for the cultivation of the inventive faculty.

The raw materials of the English mill—the seeds, nuts and kernels—come from all corners of the earth ; some are cultivated, others grow wild. They pass through so many and varied vicissitudes that the average good quality on arrival at the mill is surprising. We have no control over their production ; they are bought on sample sometimes sent from the place of origin, but generally taken on the arrival of the parcel in this country. If the sample is collected in this country, it is assumed to have been taken in accordance with a definite custom and to be representative of the bulk. It may or it may not be so, for there are several factors which have a bearing on the over-all quality of a large parcel of seed.

MATURITY OF THE SEED

If the seed is the wild-grown variety it is likely to be as varied as the blackberries on our hedges, and the native does not confine his attention to the ripe specimens. In the case of cultivated seeds, many of these (especially the small variety) are grown on small tracts of land farmed by a native, who decides very often the time of harvest not by the maturity of the seed, but the nearing religious festival, or the persistent attentions of the money-lender to whom he has mortgaged his crop.

The quantity of foreign matter in seed is very variable, and as it was not carefully distributed throughout the parcel at the place of origin the buying sample is not a reliable check. Until one has seen it done, the tendency for sand and stones to concentrate in certain bags is somewhat mystifying ; nevertheless, unless engaged in the milling industry, we cannot but admire the generosity of the native who is prepared to sell so much of his native land for a handful of seed.

Further, the seed may have deteriorated on the voyage, due to heating, method of stowing. It may have been contaminated by other cargo or have been damaged by water. The buying sample analysis is not a reliable check on mill production.

The point for a critical inspection of the seed is at the mill intake for bulk seed, and the warehouse for bag seed. Here in the case of seeds to be used for the production of edible oil is the opportunity to classify it and where necessary isolate undesirable batches. In the case of bulk seed, lifted by means of a mechanical or suction type elevator, on to a band and discharged into bins or silos, it is an easy matter to take a representative sample of each bin or silo. With bag seed it is more difficult, and the best solution appears to be to stack in definite piles. The respective silos and piles can then be classified in accordance with tests made on the warehouse samples, and seed sent to the mill can be blended to produce the desired grade of oil.

All seeds require cleaning in order to remove sand and foreign matter. Seed handled in bulk by pneumatic elevators is very free from sand on leaving the suction tube, but the more bulky foreign matter must be removed by screening. In the majority of mills the cleaning plant has to deal with all the undesirable material mixed with the seed, and it is generally placed in such position that it can operate on the seed being transferred from warehouse to mill. Small seeds, rape, til, soya beans, linseed can be very efficiently cleaned by means of the well-known revolving cylinder or reciprocating tray type of screen. Palm kernels and undecorticated groundnuts require more drastic treatment, owing to the amount of sand adhering to the skin and shell. Groundnuts taken from loamy soil are particularly troublesome in this respect, the indented shells being well adapted for holding the soil. Palm kernels and undecorticated groundnuts should be passed through a revolving inclined cylinder, sometimes termed a "tumbler," and the soil loosened by the action of the nuts rubbing together. The cylinder is sometimes perforated to allow the sand to pass out through the circumference; the more efficient arrangement is to use the "tumbler" only for the purpose of loosening the soil and to effect separation on a shaking tray screen.

The partial closing of cocoanut meat during drying forms a pocket for the accumulation of dirt, and the fine dust due to the action of weevils. This is shaken out by allowing the copra to fall over a series of steps on to a shaking tray screen.

The cleaning of woolly cotton-seed is a problem which has received much more attention in American than in this country. This is, no doubt, due to the fact that in the early days of cotton-seed crushing in this country, the seed was mainly the Egyptian variety, which has no adhering cotton and known as "black" seed. When the woolly varieties arrived the crusher was eventually faced with the problem of removing, in addition to loose sand and stones, the dirt in the lint. The evolution of the plant appears to have remained entirely in the hands of the crusher. English mills dealing with woolly seeds are equipped with cleaning plant which is more or less their own design and manufacture, or with American plant.

Woolly seed may be divided into two classes:—
1. Seed with fairly long outer lint covering a short tough fibre firmly attached to the seedhusk.
2. Seed with no long lint but covered with the short tough fibre.

To obtain the best possible yield of oil from woolly cotton-seed milled on the undecorticated system, the long lint and short fibre must be removed, the seed should be sent to the mill as bald as possible, otherwise the lint will absorb oil and the cake have a rough and, from the farmers' point of view, unattractive appearance.

Certain varieties of woolly seed, even when efficiently delinted, yield a very low percentage of oil, and as the cost of delinting is a considerable item, it requires careful supervision, as it may not be advisable to carry the process beyond a certain stage.

The lint has a market value, depending on its freedom from dust and pieces of seed hull and dirt, its length of fibre, whiteness. Apart from the length of fibre, the quality of the lint produced depends on the efficiency of the cleaning and the method of removing it from the seed. For the moment we are concerned with the cleaning of the seed and lint covering it.

The removal of loose material from the seed is easily accomplished; the difficulty is to remove the dust and dirt from the lint. Machines used for the purpose of separating loose material and removing the dust from lint are shown by the following three slides :—

Description of Cleaning Plant from Slides

1. Separator.
2. Seed and lint cleaner.
3. Arrangement of seed and lint-cleaning plant.

In the case of seed without long lint and covered with the short fibre only, the methods of cleaning described are quite efficient, but for dealing with seeds covered with long lint closely wrapped around the seed they are not efficient. It is impossible to remove the dust unless the long lint is opened out. If this could be accomplished the appearance of the lint finally obtained would be decidedly improved. The problem has in the past received much attention, but the tendency now is toward more intensive ginning and, consequently, less lint on the seed.

The removal of the lint or short fibre from the seed is accomplished in a variety of ways. For the removal of the long lint a modified form of saw gin is in general use. The Carver linter is representative of this type. The removal of the short fibre is effected by passing the seed between carborundum rolls. There are also machines of the defibrating type in which the short fibre is torn from the seed by the abrasive action of metal surfaces formed like a household grater.

Linting and Defibrating Process
Described from Slides

In England cotton-seed, having been cleaned and delinted, is sent to the mill and the oil extracted on the undecorticated system, that is, the entire seed, hulls and kernels pass through the presses. In America, where the seed is decorticated, the hulls being separated from the kernels (or meats), the meats only pass into the mill to be pressed. Much has been said and written in order to induce

English crushers of cotton-seed to introduce the decorticating system into their mills. Among the advantages claimed for the system, the following appear to be the most important.

1. It obtains a higher yield of oil.

2. The oil is of better quality than that produced from undecorticated seed.

3. The oil is refined more easily and with less loss.

4. The cake is a highly concentrated cattle food, more easily digested than the undecorticated cake.

The accuracy of items 1, 2 and 3 is generally admitted ; the yield of oil on the decorticating system being on an average $1\frac{1}{4}$ to $1\frac{1}{2}$ per cent. greater than on the undecorticated system, the comparison being made on seeds of similar initial oil distribution.

Regarding the cake, the relative merits of decorticated and undecorticated cake as a food for English cattle is still a matter of controversy. There are those who maintain that the presence of the hull is necessary to meet the conditions and methods of feeding prevailing in this country. There may be some truth in this, and the argument is not without support. The greatest obstacle to the extension of cotton-seed crushing in India is the system of feeding cattle with whole seed. The undecorticated cotton cake is not in demand, but such mills that produce it have no difficulty in disposing of the hulls to local farmers.

In this country the controlling factor appears to have been the type of cake demanded by the farmer. If there had been a growing demand for the higher priced decorticated cake, the introduction of the means of producing it would have received more serious consideration.

The plant required for decorticating cotton-seed is illustrated by means if the following slides :—

DESCRIPTION FROM SLIDES

Hullers (knife and disc).

Separating (hulls and meat).

Beaters.

Arrangement of plant.

Conditions for efficient decorticating.

Decorticating " black " seed.

Arrangement of plant for " black " seed.

During the process of decorticating linted cotton-seed, there are two sources of loss known as the separation and absorption losses. The first is due to incomplete separation of hulls and meats ; the latter is due to oil absorbed by the hulls and lint.

To obtain ideal conditions the seed should be cut in such a manner that the kernel or meat is not broken or pulverised, and the separation of hulls and meats should be effected as soon as possible. In actual practice, ideal conditions do not obtain, due to variations in

the size of the seed and wear in the huller knives and plates ; each seed is not cleanly cut, and a certain amount of pulverising takes place. This results in the production of meat flour, which adheres to the hulls and lint. Further, the seed should be cut as few times as possible, for repeated cutting, in addition to producing " flour," draws oil from the meats ; this is absorbed by the hulls and lint, and cannot be recovered. An estimation of the efficiency of the process depends on, first, the percentage of hulls mixed with the meats ; secondly, on the percentage of oil absorbed by the separated hulls. A separation of 98 per cent. meats from hulls appears to be common with modern American mills ; in fact, some claim higher efficiency. With seed properly prepared by delinting and hullers in good condition, there appears to be no difficulty in keeping the oil loss in hulls below 0·5 per cent.

It will have been noted that the system is very exacting ; it demands very definite requirements in order to obtain the highest efficiency. The American mill, which deals with seed stable in character and consistent in oil content, has no difficulty in meeting these conditions.

This is not the case with the English mill, which may have to deal with many varieties of cotton-seed, varying in oil content and character, a large proportion being " black " seed. Again, the demand for cotton-seed cake is seasonal, and during the off season it is usual for the English mill to turn to other seeds, in which event the decorticating plant standing idle would add considerably to the unproductive capital charges. It is not surprising, therefore, that the English crusher has, up to now, remained unconvinced by the arguments advanced in favour of its adoption, and after cleaning and delinting the seed sends it to the milling plant undecorticated.

All seeds should pass over a magnetic separator before milling, and in the case of cotton-seed before delinting. The most suitable place for the separator is at a point where the seed in transit is a minimum, obviously just sufficient to feed the mill. The large pieces of iron are removed by the cleaning plant, and it is the function of the magnetic separator to deal with the small pieces, which seem to have an affinity for prize cattle of fabulous value.

TYPES OF MAGNETIC SEPARATOR
Described from Slides

The subsequent operations for reducing milling, cooking and pressing the seed are interdependent, and any attempt to isolate one of them so that it does not fulfil its function in meeting the requirements of the others is soon apparent. The effect is, of course, more marked on certain seed. The effect is also cumulative for instance, unsatisfactory milling may be the cause of imperfect cooking, resulting

in excessive "foots" at the presses. In practical seed crushing there is no such thing as efficient reducing milling or cooking considered independently; the only efficiency of any use is their combined efficiency. It has already been pointed out that the method of carrying out these operations is frequently a matter of compromise—to suit the available plant. It would appear, then, that in considering the plant used in reducing milling, cooking, and pressing we should not emphasise type unduly; for there is considerable difference of opinion as to the best type of plant for dealing with a particular seed or carrying out a certain operation.

The introduction of rich oil seeds and the demands for crude edible oils revolutionised oil milling. Methods in vogue for the extraction of oil from cotton-seed, linseed, rape and other small seeds were found to be unsuitable when applied to palm kernels, groundnuts and copra. It was found that these latter required much more intensive methods, and heavier finishing rolls and presses were introduced. In the case of groundnuts and copra, the large amount of seed squeezed out of the press (foots) and the destruction of press cloths led to the introduction of the closed type of press with either fixed or movable cages. The difficulty in making a hard firm cake brought about the use of the closed press for palm kernels. The pressure applied to the cake has increased enormously—from 0·6 tons to 4 tons per sq. in., with a consequent increase in the cost of pressing plant.

During the introduction of these heavy cage presses, the gross pressures of the Anglo type press were also increased by greater ram area, and increased pressure in the hydraulic system. Further improvements in connexion with the Anglo system, such as increased cooker capacity, vastly improved cake-forming machines, together with a more intimate knowledge of the behaviour of rich oil seeds during the various processes, gave the system a new lease of life.

Opinion is now very divided as to the relative merits of the closed and open type of press. For obtaining a high oil yield from a wide range of seeds hot pressed, the Anglo system is now considered by many crushers to be even more efficient than the closed pressing system.

There is now no difficulty in producing with the Anglo press, having a gross pressure of approximately 500 tons, palm kernel cake containing only 5½ per cent. of oil. The heavy cage press with a ram pressure of 970 tons does not improve on this; in fact, if the press is filled without mats, as is usual with palm kernel, the oil left in the cake is generally nearer 6 per cent. The pressure on the cake in the open press is 1 to 1¼ tons per sq. in. and in the cage press 2·8 tons per sq. in.; the length of time under pressure is practically the same.

The press bagging in which the Anglo cake is wrapped provides a large area for the free flow of the oil. This, taken in conjunction with the fact of the full ram load being applied to the cake the whole of the time the press is open to pressure, offers a reasonable explanation for the better performance of the Anglo press. During the later stage of pressing with the cage press, the oil has to travel a very difficult path, and the pressure on the cake varies, due to the friction between the cake and the cage.

The main objection to the Anglo press for pressing palm kernel is the unsatisfactory cake produced with a pressure that will keep the cost of press bagging within a certain margin. The cake is easily broken, even after storage, and is not suitable for transit in bulk. In fact, palm kernel cake made in the cage press with a pressure of 3 tons per sq. in. on the cake is not now considered satisfactory. The latest cage pressing plant for palm kernel is arranged for a pressure of 4 tons per sq. in. on the cake and a gross ram pressure of 1300 tons.

The utility of such a plant for other seeds for instance, ground nuts and copra, is questionable, for these do not require such intense pressure for the production of firm cakes. In fact, in the case of ground nuts it is in the home trade a frequent complaint that cage press cake is too hard.

Here again (dealing with ground nuts), the cage press requires a greater pressure per sq. in. on the cake than the open press for equal oil yield, and the troublesome "foots" associated with decorticated ground nuts are not entirely eliminated by the use of the closed press. It is very difficult in the case of heavy cage presses with large ram area to regulate the speed of the ram, to keep the quantity of oil drawn within the capacity of the cage drainage. The trouble occurs during the early stage of pressing, and if the ram is allowed to rise too rapidly more or less meal is washed out of the top and bottom of the cage. This restriction in speed of working together with the use of mats reduces the output of ground nuts considerably below that of palm kernel. The increase in size (due to wear) of the perforations which form the drainage area must also be taken into account, for as these increase in area so does the quantity of "foots." In the case of plate cages the perforations are usually 0·03 in. dia., and an increase to 0·04 in. makes an appreciable difference in the quantity of "foots," and the labour required to keep the cage chambers clean.

For dealing with rich oil seeds, and for cold pressing, the cage press has decided advantages, but for dealing with a wide range of seeds and producing a high oil yield, the Anglo or open type press, with its simplicity and low upkeep costs, is by no means supplanted. It is yet the utility plant of the mill.

The highest possible yield of oil is not the only consideration in seed crushing; the production of cake that will satisfy the requirements of the farmer must also be taken into account. The average farmer is now quite alive to the value of oil in the cake he uses, and that cake containing a high percentage of albuminoids and a low percentage of oil is not desirable. Further, he places particular value on the kind of oil, for instance, linseed and cotton seed, linseed cake being certainly the most popular, mainly due to its high oil content.

Cakes are now sold guaranteed to contain a certain percentage of oil, and actually the oil content is higher than the guarantee.

The oil content of the most important straight feeding cakes is given below :—

Linseed cake	..	..	7–8%
Cocoanut cake	..	..	$6\frac{1}{2}$–8%
Groundnut cake		..	7–8%
Palm kernel cake		..	$6\frac{1}{2}$–8%
Cotton seed cake		..	5–6%

Owing to the amount of oil required to be left in the cake made from certain seeds, it is neither necessary nor the practice to make extraction as intensive as possible, and in the case of the open press the length of time the cake is under pressure is reduced, with a corresponding increase in output. Open type presses, arranged four presses per " side " or battery, are usually filled and discharged at the rate of six pressings per hour from the four presses, when the highest oil yield is desired. For the production of cake to contain a higher percentage of oil, seven or eight pressings per hour are taken from the four presses. This statement, however, requires some qualification ; for in the case of rich oil seeds it is not possible to work the open press at the higher speeds, owing to excessive " foots." The open press does not then compare so favourably with the cage press, as will be more fully shown in the following.

It is obvious, then, that in considering the detail operations in connexion with reduction, milling, cooking and pressing, it will be more to the point to emphasise what effect each operation should have on the seed ; and how the desired effect may be obtained. We shall endeavour to do this during the description of the following slides :—

REDUCTION MILLING

Cooking—pressing—" foots."

EXTRACTION OF OILS BY MEANS OF SOLVENTS.

For many years the pressing or expression system stood alone in this country as the only efficient means of obtaining oil from seeds. The cake gradually became established as a cattle food, and the farmer is now quite familiar with the residue of certain seeds in the form of cake. His system of cattle feeding is built up on its use. Further, many of the standards of value for oil and cake were based on the capacity of the pressing system, and have been revised as the system improved.

When the solvent system of extraction was introduced, one of the most difficult problems which had to be solved was the disposal of the residue, which in the form of meal contained much less oil than the standard cake made from similar seed. The increasing demand for compound feeding stuffs solved the problem—the solvent plant meal became a recognised ingredient in the manufacture of sweetened meats, compound cakes, cubes, etc. At first it was used very sparingly owing to the prejudice against its use on account of low oil content and frequent trace of solvent. Improved methods and the use of more suitable solvents facilitated the production of solvent free meal, and the value and use of a low oil content ingredient for the compound mix is now fully appreciated.

The increasing demand for meals, etc., also brought about the extraction of oil from a larger variety of seeds by the solvent method, and although it is not suggested that the solvent plant meal will entirely take the place of cake meal in the compound mixtures, it is being used in increasing quantity. Further, there is a decided tendency to produce meals of such oil content that will permit of there being used more freely than heretofore. As the guaranteed oil content of compound feeding stuffs is fixed much higher than that in the usual solvent plant meal, the quantity which can be mixed with other ingredients of normal oil content is obviously limited.

The solvent system has developed rapidly since the war. There are now several plants each dealing with close on 1500 tons of seed per week. Undoubtedly (allowing for wage fluctuations common to both systems) the cost of extraction by the solvent method has been considerably reduced during the past few years. This has been brought about by increased production per unit (usually considered as 4 pots) reduced solvent losses, cheaper solvent and less consumption of steam.

Owing to the fact that the system lends itself to increased output without labour interference, there is every incentive to a still further increase per plant unit.

For the production of crude edible oil, the question of a suitable solvent is important. It has been proved by experience that for the higher quality oils, the solvent must comply with certain definite requirements. The unsatisfactory solvents and crude distilling plants used in the early days did much to retard the progress of this system of oil extraction.

For the extraction of oil from seeds a solvent should contain no traces of any compound capable of undergoing chemical change during the extraction process. It should have a close range of distillation, not too low to encourage loss, nor so high as to require a final temperature of evaporation injurious to the oil. It should

not be soluble in water and it should have a distinctly different gravity from water. A table showing the characteristics of some of the available solvents is shown on the slide.

SOLVENTS.

Solvent.	B.P. (°C.).	Sp. Heat.	Latent Heat cal.-gram.	Price per gall. s. d.
Ether	34·6	·53	90	4 0
Carbon di-sulphide	46·2	·24	83·8	3 7
Chloroform	61·2	·23	58·5	25 2
Alcohol (methylated spirit)	78·1	·6	205	3 1
Carbon tetra-chloride	76·7	·2	51	6 2
Benzol (pure)	80·3	·41	93·4	1 7
Benzol (commercial)	79–125	·408	95	1 3
Trichlorethylene	87	·233	56·6	5 3
Petroleum spirit	100–120	·425	73·5	1 6
Toluol (pure)	110·8	·404	—	1 10
Turpentine	·59	·41	74	4 10

For general suitability there has up to quite recently been nothing comparable to the specially prepared petroleum spirit. It has a satisfactory boiling point, and a sufficient close range to place it on an equivalent with the constant boiling solvent. Further, the standard grade which has been proved to be suitable for the extraction of edible oils, can be obtained at a reasonable price. Unfortunately, it is inflammable, and its use adds considerably to the insurance charge on plant and buildings, especially in a congested district and owing to the special precautions which have to be taken in the arrangement of artificial lights it is difficult to obtain good plant illumination.

Within recent time, trichlorethylene has come into prominence, for the extraction of oil from seeds, and it would now appear to be conclusively proved that in addition to being non-inflammable, possesses in use the following advantages compared with petroleum spirit (benzene) :—

It has greater penetration.
Less solvent losses.
Increased production per plant unit.
Lower steam consumption for evaporation.

Regarding the use of this solvent for the production of edible oils, there is considerable difference of opinion as to its suitability ; this can only be decided when the solvent comes into more extended use.

The high cost of trichlorethylene compared with that of benzene is against it coming into rapid general use for oil seeds. The cost of solvent with plants using benzene is not now such a serious matter as formerly, when losses were anything up to 7 gallons per ton of seed ; and solvent at 2s. 6d. per gallon. The modern plant with a solvent loss of 3 gallons (benzene) per ton of seed and obtaining solvent at 1s. 6d. per gallon is not likely, on the face of it, to look very favourably on trichlorethylene at 5s. 3d. per gallon. To arrive at a fair comparison the conditions appertaining to the particular plant likely to use it must be taken

into account. Further, in order to make perfectly sure that full benefit may be derived from the higher working efficiency of trichlorethylene, the capacity of the plant details must be checked.

It is frequently stated that more patent specifications are drawn up for rotary pumps than any other appliance, the number for solvent plants must be a good second. It is astonishing how few of these patents have been put into actual working. The type of extractor pot used appears to be fairly common —and has a capacity of 2 to 2½ tons of seed. It is stationary and the meal is agitated by means of stirrers fixed to a vertical shaft.

In other details of the plant there is considerable variation. There are also several methods of washing the seed with solvent.

As no useful purpose can be served by considering old abandoned designs, we shall confine our attention to the modern plant and with the help of the following slides describe the plant details and methods of carrying out the several operations in connexion with the process.

DISCUSSION.

In opening the discussion, PROFESSOR HINCHLEY made reference to the pressure of 4 tons to the sq. in., which he thought higher than was necessary for the best return for the money involved and the labour and material used.

Regarding solvent extraction, Professor Hinchley thought it was bound to knock out press extraction, but unfortunately for industries these changes take place slowly. It was obvious that press extraction was getting to its limit on account of the fact that these high pressures were being reached and capital losses caused. The cost of solvent extraction was very small, possibly not more than one-fifth that of press extraction in a well-conducted scientifically-run plant. It wanted the application of still more chemical engineering to diminish it further.

The use of trichlorethylene in these plants was a question which Mr. Brewis raised and which he (Prof. Hinchley) would like strongly to urge. He thought the use of heavy solvents gave a method of extracting oils at a price that was incomparably cheaper than any other method. Unfortunately, trichlorethylene had a habit of decomposing and a trace of hydrochloric acid appeared ; it was, however, unlikely to give very much trouble.

Dealing with the question of the residue being used for cattle food, Prof. Hinchley remarked that there was another use for these residues. In the case of poisonous seeds, it was obvious that it could not be used as cattle food, but with the addition of 5 to 8 per cent. of caustic lime a material was formed which made an excellent floor-covering and excellent tiles.

Dealing with the question of size of meal, Prof. Hinchley said in his opinion all the people who worked on solvent plants crushed the meal too much. He considered that pea-size was quite small enough.

Concluding his remarks, Prof. Hinchley said in regard to edible oils, he felt that the right place, generally speaking, to deal with them was where the nuts were produced. The fresher the seed the better the oil produced, and it was quite possible that this industry would develop in the countries where the seeds were produced.

Mr. J. W. Austin said that, as a Member of the American Institute of Chemical Engineering, he would like to mention some of the differences met with in America. In the first instance they could not get so good a solvent as in England. Their solvent commenced to distil at about 90° F., and had a boiling point of 320° F.; it was very rarely that they got anything under 300° F. The consequence was they were troubled in getting that close range to which Mr. Brewis referred. One way to deal with it was by a fractionating still. Where possible, benzole was used instead of benzine or trichlorethylene, but it was not possible to use benzole on all substances; for instance, on cotton seed it dissolved some of the colouring matter. The proper solvent to use was benzine.

With reference to the use of trichlorethylene, it was true that the makers of that commodity said that it could be used without decomposing, but when the matter was put up to them asking them to guarantee that, they would not do so.

The manufacturer of solvent extract plant had in some measure to guarantee his plant, and consequently had said to some of the U.S. makers of trichlorethylene that until they were in a position to guarantee that it would not decompose, he (the manufacturer) preferred not to handle it.

Another difference was the climatic conditions. In America there was a much drier air than in England, and also, particularly in summer time, a much hotter one. This affected the possibility of condensing the solvent vapours, particularly if benzine were being used, which had a low boiling point fraction. Climatic conditions were also important because in some cases it was very easy to generate static electricity with belts running at a high speed, and that was rather dangerous in a solvent-extracting plant where it was impossible to avoid vapour in the building where the plant was enclosed. That had been dealt with by grounding the shafting, but another way was to use a condensing rope which would give a lower speed in some instances; it also was more easy to ground than the belt.

Perhaps the greatest difficulty in interesting American manufacturers in solvent-extracting plant was that nearly always they wanted to deal with such large quantities, and in order to get efficient extraction it was far better to deal with the material in small quantities.

thus making it possible to bring the material being extracted and the solvent used for extracting, the steam and the water for condensing into more intimate contact. In America the extractor such as had been shown was called the English type, and the American type was called the Rotating Extractor: it was a large vessel rotating either on two trunnions one on each end, or by supports round the body of the vessel. It would hold, of course, a much larger charge than the pot system, but immediately a large charge was dealt with any number of difficulties arose. In fact, he (the speaker) knew of several plants which sought to deal with edible products in this way, which were a complete failure, owing to the impossibility of dealing with the material efficiently.

Mr. W. Ramsay Sibbald referred to the difficulty of avoiding the production of flour when dealing with ground nuts. It appeared to him that the first essential, especially in extraction process, was to break the seeds open. In the case of ground nuts, the material was almost unique. Oil-bearing seeds contained starch, and immediately the cells were broken open, and particularly when attacked by solvent, there was a tremendous outpouring of very minute starch grains.

Mr. J. A. Reavell (Chairman of the Chemical Engineering Group) asked for information about trichlorethylene, which was supposed to be nonflammable. He knew of two occasions in which explosions and fires had occurred. In one case, trichlorethylene had been used in connexion with bones. Several theories had been put forward, and he would like to ask whether the lecturer had himself come across that difficulty.

Another point upon which he would like information was the nature and extent of the losses mentioned in this solvent process and where exactly these losses occurred.

Mr. J. Savage said that in the early stages of the manufacture of trichlorethylene, decomposition was rather severe, in some cases due to the product being impure. In the early days certain difficulties had arisen and had been successfully surmounted, and now very pure product was obtained. Recent research had brought out certain improvements in the process, with the object of eliminating decomposition.

Regarding the explosions mentioned by the last speaker, he knew of one at Roubaix, in he believed 1912. It was thought to be due to trichlorethylene, but was afterwards proved to be due to a blockage. He had not heard of an explosion in connexion with bone-extraction plant.

Mr. Austin asked whether the makers of trichlorethylene were aware that moisture was present in the raw material, and that some steam had to be used in the recovery process to carry off the solvent from the extraction, from which it would appear impossible alto-

gether to separate the moisture from the trichlorethylene.

Mr. SAVAGE said that decomposition of trichlorethylene was not due to the presence of water, but was due to actinic rays. The admission of light in any way effected decomposition, especially in the presence of air or oxygen.

Mr. BREWIS, replying to the discussion and to a vote of thanks, moved by Colonel Brigg (who had taken the chair when Mr. Thompson had been compelled to leave), said he agreed with Prof. Hinchley that in the extraction of oil from seeds, many alterations both in process and type of plant employed would be made in years to come. He hoped he had not been misunderstood when speaking of a pressure of 4 tons per sq. in. on the cake. This pressure was applied in a cage type of press lately introduced for the production of cake for export. It had been found that palm kernel cake manufactured under the usual pressure, and exported in bulk, was broken at the end of the voyage. The ordinary type of cage press making $18\frac{1}{2}$ in. cakes had now been reconstructed to make cakes $16\frac{7}{8}$ in. square.

He further agreed with Professor Hinchley that meal to be extracted by the solvent process was generally reduced to too fine a degree and the use of trichlorethylene enabled full advantage to be taken of this, resulting in a considerable saving of rolling power.

He hoped he had not conveyed the impression that the stirrers were put into operation while a charge of seed was being washed with solvent. This was quite against modern practice, and if done during the process of extraction considerable difficulty would be experienced in finally dealing with the charge. The use of the extractor was for breaking up zones or pockets during the process of steaming-off.

Speaking of references he had made to the losses which occurred in extraction plant, he said it was well known that formerly plants were constructed with solvent-saving devices, and that they were worked in such a manner that there were more displacements of air in the extractors than had since been found necessary. To keep the solvent losses as low as possible, it was absolutely necessary to work the plant on such a system of washing that the displacements of air could be kept at a minimum.

The system of simply allowing the air to pass through a water bubble tank was out of date, air being now treated in a much more efficient and reliable manner.

Referring to the risk of fire with trichlorethylene he had never investigated this, but he believed that trichlorethylene would go a long way. It was an established fact that plants were being operated with it without experiencing any trouble from corrosion, and were receiving full advantages from the greater penetration, lower steam consumption and other items to which he had referred in the paper.

THE MANUFACTURE OF POTASH AND OTHER SALTS FROM LEUCITE.

By J. W. HINCHLEY, Wh.Sc., A.R.S.M., F.I.C., M.I.Chem.E.

It is a disquieting fact that the whole of the world's supply of potash is still obtained from practically one district (95 per cent. comes from Strassfurt, Germany, and 5 per cent. from Alsace). This is a matter of concern, not only to the British Government, but to the United States, Italian and other governments, and in these countries diligent outlook is kept with a view to obtaining equally cheap supplies from other sources.

The most hopeful source for supplies would appear to be the igneous rocks, which contain, on an average, 3 per cent., while some contain as much as 10 per cent. of potash. It is obvious that these igneous rocks may present some chemical difficulty in attack, as compared with the soluble salts of Strassfurt (sylvine, carnallite, etc.). In the case of these soluble salts of Strassfurt, a complicated and somewhat costly process of crystallisation has to be carried out to obtain pure products, but for agricultural purposes a crude product is usually satisfactory. If, however, pure minerals may be obtained from igneous rocks which are readily treated by chemical methods, it may be possible by these means to compete with the soluble salts of Strassfurt.

Of those minerals which occur in igneous rocks leucite is the most attractive. It is a comparatively rare mineral, except along the line of the Italian volcanoes.

It has some historical interest from the fact that Klaproth discovered potash in 1796, through examining it. The millstones of Rome have been made from leucitic lava for over 2000 years.

The use of leucite as a raw material for the manufacture of potash salts would bring another country into the potash market, and certainly relieve the world's situation with regard to potash, and possibly lead to the utilisation of similar or other minerals occurring in igneous rocks in other countries. Leucite (K_2O, Al_2O_3, $4SiO_2$) in a state of purity contains 21·5 per cent. of K_2O, 23·5 per cent. of Al_2O_3, and 55 per cent. of SiO_2. As a rule the natural mineral contains less potash, on account of the presence of magnesium and sodium, but this replacement of potash and other elements varies among the different volcanoes, and in some places, practically pure leucite occurs in extraordinarily large quantities.

Orthoclase is another mineral occurring in igneous rocks, which offers possibilities for the extraction of potash. It occurs in smaller proportion than leucite in the lavas of the Italian volcanoes, and, unfortunately, is usually associated with albite (soda felspar), so that whilst pure orthoclase contains 16·9 per cent. K_2O, 18·4 per cent. Al_2O_3, 64·7 per cent. SiO_2, that occurring in the Italian lavas contains only 7·1 per cent. of potash.

In a few of the Italian lavas the amount of orthoclase exceeds that of the leucite; in a great majority it is much less, but in a large number of leucitic lavas, orthoclase is entirely absent.

Mr. Henry S. Washington has made a large number of analyses of the Italian leucitic lavas, and states that the average potash content is 8·72 per cent., whilst the average for all lavas, leucitic and non-leucitic, is 8·31 per cent. of potash. A region of igneous rocks so high in potash is unknown in any other part of the world.

The row of volcanoes extending 186 miles from Orvieto to Naples, contains seven craters, of which Vesuvius alone is active, the others being Bolsena, Vica, Bracciano, Alban Hills, Hernican Volcano, and Rocca Monfina. From a careful examination of the rocks during the war by Baron Blanc, it was found that the leucitic rocks of Rocca Monfina contain the purest leucite, the leucite being practically free from magnesium and sodium. The leucites of other volcanic regions contain in many cases considerable quantities of these elements.

The author has examined the leucitic rock obtained in the Rocca Monfina district, at places about half-a-mile apart, and has found that the composition of the leucite is practically the same.

The Rocca Monfina volcano lies about thirty miles to the north of Naples. Its igneous rocks cover an area of about 80 square miles, and are computed by Mr. Henry Washington to contain 1008 million metric tons of K_2O. There are some flows of non-leucitic lavas—basalts, andesite, and trachyte—but the leucitic lavas are the most abundant. The average content of potash in the latter is about 10·5 per cent.—the highest figure for K_2O in the whole of the Italian lavas. Occasionally on the surface the leucite is somewhat kaolinised, but this is the exception rather than the rule. At the centre of the volcano is a small cone, Monte Santa Croce, of non-leucitic andesite. It would appear that this

volcanic district could supply the world with potash at the present rate of consumption for a thousand years.

The leucitic lavas which are being worked at the present time at Fontanaradina are about twelve miles from the little port of Scauri. The rock, after removing a very shallow layer of soil, is at once capable of being quarried, and by a simple telpherage system can be carried straight down over the intervening country to the wharf at Scauri by gravity. Fontanaradina lies about 370 metres above the intervening country, lying towards Scauri.

A photograph of a section of the rock is shown in Fig. 1 ; × 12. The regular trapezoidal form of the crystals is clearly shown. Examined under polarised
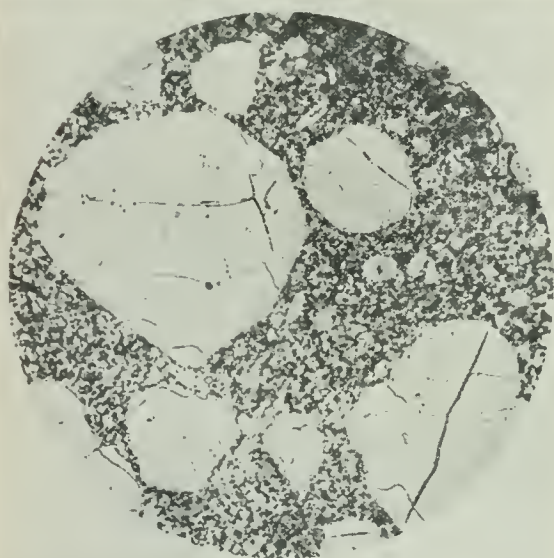
Fontanaradina, Rocca Monfina, Italy
Section of Leucitic Lava × 12

FIG. 1

light the crystals are found to be under stress ; when heated to 500° C., however, this stress disappears and the simple crystalline form is revealed. Inclusions of gangue are very common in the leucites of many of the lavas, but this is not so common in those of Rocca Monfina.

A view of one of the workings is shown in Fig. 2, from which the simplicity of the mining will be seen.

It will be obvious that unless the leucite can be separated from the gangue, the proposition of the manufacture of potash salts at a reasonable cost may be practically impossible. It has been found, however, that the small content of iron in the gangue is sufficient to permit of perfect electro-magnetic separation of the two materials.

The leucite occurs in the rock in crystals, varying from 3 in. in diameter to microscopic dimensions,

but after crushing the rock and sifting to obtain uniform granular material, a very simple machine, which has been devised by Baron Blanc, brings about at a very rapid rate the complete separation of the leucite crystals from the gangue.

The mill in which the crushing, sifting and electro-magnetic separation takes place, is shown in Figs. 3 and 4. The rock is crushed in jaw-breakers and rolls, sifted to separate into granular material and fine powder and then treated in " belt " electro-magnetic separators.

For chemical treatment it is desirable, for reasons which will appear later, to use material whose grains are about one-sixteenth of an inch in diameter.

The fine powder produced during the process of crushing may be used as a potash manure.

Dr. Voelcker has carried out a series of experiments during the last three years, on the value of powdered leucite as a manure, and has found as a general conclusion, from his experiments, that the value of leucite is practically identical with that of soluble manures of the same potash content. His pot experiments may be cited :—

TABLE VI

Treatment	Weight of corn g.	Weight of straw g.	Percentage of untreated Corn	Straw
1. Untreated	8·19	16·70 ..	100	100
2. Leucite—5 cwt. per acre	11·23	19·99 ..	137	119
3. Sulphate of potash containing potash equal to 5 cwt. per acre of leucite	11·00	18·55 ..	134	111

The chemical properties of leucite are extremely interesting, and there is little doubt that as it becomes better known it will be used for many purposes and in many processes in which it is at present unknown. The potash present is readily replaceable by many other elements. It is related to the zeolites, but contains no water. Its application to water softening and similar purposes is obvious.

The behaviour of natural silicates with acids varies enormously. In some cases the attack is nil, and in many cases in which attack takes place gelatinous silica is formed, and the separation of the liquids from the solids becomes extremely difficult, and may make the cost of treatment excessive and impossible. On treating powdered leucite with acids, solution takes place with evolution of heat and gelatinous silica is formed in sufficient quantity to make the process difficult on a commercial scale. If, however, granular leucite is treated with acids the amount of gelatinous silica formed is small, but still sufficient to seriously interfere with economic manufacture. Baron Blanc, however, found that if the acid liquid be continually circulated through the granular leucite a solution was obtained in which no gelatinous silica occurred. The gelatinous silica which must be formed through the presence of small quantities of powdered leucite is removed from the solution by the filtering or adsorbing action of the granular material. At the

end of the process of solution in acid the crystals of leucite appear as little skeleton crystals of silica.

The granular material obtained by the electro-magnetic separation of the crushed rock contains :—

K_2O	..	..	18–20 per cent.
Al_2O_3	.	..	22–24 ,,
SiO_2	..	..	53 ,,

On examination of the crystals of leucite separated, there will be found inclusions of gangue, so that many of the grains consist partly of leucite and partly of gangue. On this account the separated material usually contains from 2 to 3 per cent. of gangue.

action of sulphuric acid on the material according to the equation :—

$$K_2O,Al_2O_3,4SiO_2 + 4H_2SO_4 + 20H_2O = $$
$$K_2SO_4, Al_2(SO_4)_3, 24H_2O + 4SiO_2$$
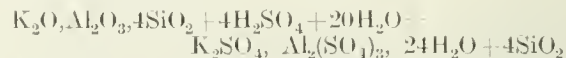
A plant treating 4 tons of leucite per day by this process is in operation at Pierre Benite, near Lyons, France. It yields 6 tons of alum per day.

Granular leucite is treated with circulating dilute sulphuric acid which is continually enriched as combination takes place. The heat of solution of the acid in the water and that due to the forma-tion of alum raises the temperature of the liquor and the action becomes vigorous. By the circulation

One of the Quarries

FIG. 2

On treatment of the separated material with acid, a certain amount of iron enters into the solu-tion, but the amount of iron is not sufficient to interfere with the chemical processes, or to affect seriously the purity of the chemical substances produced by very simple processes.

MANUFACTURE OF ALUM

Perhaps the simplest industrial process is that of the manufacture of alum and silicate of soda from this material. Potash and alumina are present in the correct proportions for forming alum by the

of the liquor through the granular mass, any colloidal silica produced at the beginning is absorbed by the silica formed, and a solution is obtained which is free from silica but contains a little iron.

It is well known that iron is a serious impurity in alum, and therefore it is essential that this iron should be completely removed from the final pro-duct. The gangue is almost unattacked by acid even when hot, so long as it has not been reduced to powder. In actual practice saturated mother liquors are used for the solution of leucite, and when the process is complete the temperature has been raised to about 95° C. On cooling, alum

corresponding to the amount of leucite attacked, crystallises out, and is separated and washed in a centrifuge, by which a very pure alum is obtained. During this crystallising process the liquor should be agitated so that the crystals are produced in micro-crystalline form ; as a result the whole of the iron remains in the mother liquor, and the crystals are obtained in the form known as fine crystals. About 20 per cent. of the alum present in the solution is crystallised out on each operation.

The mother liquors are used for a further treatment of the mineral, but as a rule the iron present

serve at once for the formation of important silicon compounds. As obtained in a dry state it usually contains about 18 per cent. of water, corresponding to $4SiO_2, 3H_2O$. On drying in a vacuum desiccator at the ordinary temperature this is reduced to $4SiO_2, H_2O$; on being exposed to moist air, it again takes up water. Its properties correspond therefore with those of ordinary precipitated silica.

MANUFACTURE OF SILICATE OF SODA, ETC.

On adding this silica to solutions of caustic soda or potash, even when very diluted, solutions of corres-

Works seen from the Road

FIG. 3

is removed by simple chemical treatment, either in neutral solution by precipitation with a small amount of ammonia, so that a small precipitate of aluminium hydrate with ferric hydrate is formed ; or, in acid solution, by adding a small amount of milk of lime or baryta to serve the same purpose. Agitation should be continued during this precipitation. On account of the large amount of water removed as water of crystallisation, the mother liquors, after being freed from iron, are diluted and again acidified and used for the treatment of a new batch.

The residual silica after washing with water is removed from the dissolving apparatus, and on drying becomes reduced to an impalpable powder, and by a simple air separation gives a very pure product. This silica is remarkable in that it may

ponding silicates are almost immediately obtained. The reaction takes place according to the equation :—

$$2 NaOH + nSiO_2 = Na_2O, nSiO_2 + H_2O$$

Such silicates of soda and potash, when the air-separated silica has been employed, are extremely pure and may be obtained of practically any composition. Commercial silicates may be made by using the wet residue from the manufacture of alum. The gangue is not attacked by caustic soda or caustic potash, and is separated from the silicate solutions formed by settling.

The importance of this process of making silicates will appear when one compares it with the usual method of manufacture. It avoids the fusion of silica with the solid alkali and the subsequent treatment in an autoclave of the solid silicate obtained.

The composition of commercial silicate of soda is (40° Bé) :—

SiO$_2$	..	..	..	26·1 per cent.
NaOH	..	..	..	9·8 ,,
Water	..	..	..	54·1 ,,

The residual silica from Rocca Monfina leucite usually contains 5 per cent. of gangue and 40 per cent. of water. It is therefore a simple matter to calculate the amount of caustic soda solution of given composition to add in order that commercial silicate of soda (40° Bé) may be obtained.

The reaction is spontaneous and rapid; the caustic soda solution is placed in an ordinary vessel provided with an agitator, and the silica residue is gradually added. The reaction is exothermic, so that there is a rise in temperature. The operation may be accelerated in the beginning by warming the caustic soda solution. In practice, the silica is usually added slightly in excess, and while the liquor is hot it is rapidly pumped through a filter press, from which the silicate of soda of 40° Bé, issues, pure, colourless and transparent. The proportions used in practice amount to 474 kg. of silica and 549 kg. of soda of 29° Bé to obtain one ton of 40° Bé silicate of soda. The cost of production by this process in France with sulphuric acid at 22 centimes per kg. is 31 centimes per kg. for alum and 18 centimes per kg. for sodium silicate, or in round figures, with acid at £3 per ton, the cost of production of alum is £4 4s. per ton and of sodium silicate £2 19s. per ton.

The production of alum and silica of soda at Pierre Bénite corresponds to an 80 per cent. yield.

Hydrochloric Acid Treatment of Leucite

The treatment of leucite with hydrochloric acid presents greater chemical engineering difficulties than the treatment with sulphuric acid, on account of the volatility of hydrochloric acid, and also of its greater corrosive action. The material is treated in a similar manner, as in the former case, but the plant must be completely closed, with a vent connected to a suitable scrubbing tower for the removal of hydrochloric acid fumes. This scrubbing tower may be packed with leucite with advantage, the water which trickles down it being used in due course to dilute the mother liquor obtained in the operation. Mother liquor in suitable quantity (usually from 2 to 2½ cubic metres of liquor per ton of leucite treated) is continually circulated through a bed of granular leucite, and gradually acidified, as solution takes place. The circulation of the liquor is brought about by a centrifugal or other pump, and the liquor meets at one point in the plant hydrochloric acid gas, so that as attack takes place further acid is provided. For a reason which will be realised presently, strong acid solutions may reduce the rate of solution. In the course of 2 to 4 hours the whole of the potash and alumina has gone into solution and the temperature has risen to a little above 90° C. The amount of heat generated by the process is so great that the plant must be arranged to dissipate a proportion of it or the temperature may rise too high.

Since the solubility in the cold of aluminium chloride is much greater than that of potassium chloride (400% Al$_2$Cl$_6$ 32% KCl); and since the solubility of potassium chloride is nearly twice as great at 90° C. as at 15° C., if suitable quantities are chosen, the whole of the potassium chloride dissolved in the reaction may be crystallised out, by cooling the resultant liquor obtained. In practice the amount of solution used is such that it is saturated with potassium chloride at a temperature not higher than 70° C. After discharge from the dissolver, the liquor is passed into a cooling apparatus, in which the potassium chloride crystallises out. Evaporating coolers are very convenient for this purpose, since the crystals form on the surface of the liquor, and the operation need only take a very few hours. About one third of the potassium chloride present in the liquor is crystallised out in cooling.

The magma of potassium chloride crystals and concentrated solution of aluminium chloride are run into a centrifuge, in the basket of which the potassium chloride is retained, and is washed with a small quantity of water, or potassium chloride solution, by which white crystals of 98/99% purity are obtained.

The aluminium chloride solution is now treated in a closed vessel with hydrochloric acid gas, when crystals of hydrated aluminium chloride are precipitated; the rise of temperature which takes place during this operation through absorption of hydrochloric acid is useful as it prevents any risk of further crystallisation of potassium chloride. At this point, water may be added to the liquor with advantage, to restore to the liquid some of that removed by the crystallisation of the aluminium chloride. The concentrated solution of hydrochloric acid and mother liquor thus obtained is transferred to the dissolver, whilst the precipitated hydrated aluminium chloride is transferred to a porous brick filter, washed with water, and drained.

By this method of operation, practically the whole of the potassium and aluminium present in the leucite is obtained in the form of potassium chloride and hydrated aluminium chloride. The process is one which needs careful attention to detail; it is obvious that the liquor at the end of the dissolving stage will be acid, and that there may be risk of precipitation of aluminium chloride on this account, but practical experience has shown that some time is required after the end of the attack on the leucite, before the passage of the hydrochloric acid gas can give that degree of concentration which is necessary for the precipitation of hydrated aluminium chloride. The mother liquor rejected from the last operation will obviously be nearly saturated with potassium chloride, and it is obvious that if it is used for the attack on a fresh supply of leucite, the whole of the potassium in the mineral treated may be crystallised out by cooling the liquid. It is found that the volume of liquor needed for each operation is such as will contain about three times the amount of potassium chloride corresponding to that in the charge of mineral.

It will therefore be realised that this constitutes a simple cycle of operations, by which the same mother liquors are returned to the cycle continually, and any impurities may be concentrated in these mother liquors.

Ferric chloride accumulates this way, and tends to discolour the salts obtained, although simple washing easily removes it.

The author examined the results of a series of ten operations carried out on a small scale, and it appeared that the colour of the solution due to ferric chloride was practically the same in each case, a

of aluminium when heated to about 280° C. decomposes according to the reaction :—

$$2AlCl_3,6H_2O = Al_2O_3,3H_2O + 6HCl + 6H_2O.$$
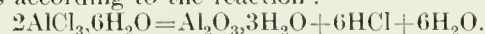
This reaction is carried out on a practical scale in a tubular furnace. The inclined rotary tube may be made of aluminium or of iron, since neither is attacked by hydrochloric acid at this temperature, even in the presence of water vapour. The hydrochloric acid thus produced serves for attacking a fresh charge of leucite, but it must be observed that it is associated with an equal amount of water ; on the other hand also, for the purpose of ensuring that the

Works, with Rocca Monfina in the Distance
Fig. 4

point being reached in the second or third operation by which the amount of iron eliminated with the chlorides of potassium and aluminium, is equal to that entering the solution with each new attack. This state of impurity of the solution gives no cause for trouble. Although the iron may be readily removed in practice, it is found that there is no necessity for this, provided that the crystallisation of the two salts takes place while the liquid is in a state of agitation. The crystals which will form under these conditions are very pure and free from ferric chloride, except as wetted by the solution, and a small amount of washing water readily removes this.

The aluminium chloride obtained, may be further treated, so that hydrated oxide of aluminium is produced and hydrochloric acid driven off. This process depends on the fact that hydrated chloride

aluminium hydrate is free from acid, an additional amount of steam may be introduced at the discharge end of the rotary tube. The alumina usually produced contains less water than the equation indicates —the amount corresponds to $Al_2O_3, 1\frac{1}{2}H_2O$. The hydrochloric acid thus driven off, on being admitted to the dissolving plant, will play its part in the attack of the leucite, while the water vapour will pass off to the scrubbing tower connected with the plant.

Quantities of Materials

It is now worth while considering the relative quantities of materials required for carrying out this process :—618·3 kg. of hydrochloric acid gas are required to attack one ton of leucite (19 per cent. K_2O, 22 per cent. Al_2O_3), of which 471·1 kg. will form aluminium chloride, and 147·2 kg. will form potassium chloride. The 471·1 kg. fixed by the aluminium would, of

D

course, be liberated if that aluminium chloride were transformed into aluminium hydrate. Should potassium sulphate be required, the displacement of chlorine from the potassium chloride by well-known methods, would mean that 197·6 kg. of sulphuric acid would be required per ton of leucite treated ; the amount of liquor required is about 2500 kg. per ton of leucite. The reaction which takes place is the following :—

$$K_2O,Al_2O_3,4SiO_2+8HCl=2KCl+2AlCl_3+\\4SiO_2+4H_2O.$$
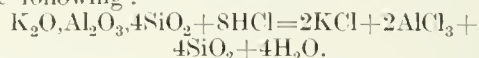
A small amount of water is thus produced by the reaction, but in a well-closed plant, the loss of water by evaporation during the process of solution, is sufficiently great to necessitate the addition of water. The water produced by the reaction is found to be associated with the silica, apparently in a loose state of combination.

Each molecule of aluminium chloride precipitated is associated with six molecules of water, as water of crystallisation $(AlCl_3,6H_2O)$, so that in the treatment of one ton of leucite, 466 kilos. of water will be taken up in this way. The quantity of water required for washing the silica is about 200 kilos. per ton of leucite treated, and is again used for washing the chloride of potassium and chloride of aluminium obtained.

Heat Equations

In the attack of leucite, we have three exothermic reactions which serve to bring and keep the liquid at the temperature of ebullition when one works with considerable charges of material. The reactions are as follows :—

(a) $K_2O+2HCl=2KCl+H_2O+102·3$ cals.
(b) $Al_2O_3+6HCl=2AlCl_3+3H_2O+67·2$ cals
(c) $HCl(gas)+nH_2O=17·3$ cals.

For one ton of leucite, it would appear, therefore, that by reaction (a) 227,253 calories are produced, and by reaction (b) 118,746 calories. With regard to the reaction (c), the total heat produced may be taken to be about 293,000 calories for each ton of leucite treated. It will thus appear that 600,000 calories are generated by the attack of hydrochloric acid gas on one ton of leucite—a quantity of heat more than is required to bring and keep the liquid at the boiling point. It is therefore necessary to provide means in the plant of dissipating heat, and to conduct the reaction so that a temperature of over 90° C. is not obtained.

The decomposition of hydrated aluminium chloride in the tubular furnace is an endothermic one.

(d) $2AlCl_3,6H_2O+211·4cals.=Al_2O_3,3H_2O+\\6HCl+6H_2O$
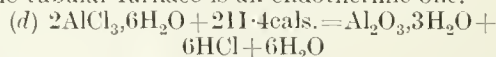
It will therefore be necessary to provide 2070 calories for each kilogramme of aluminium hydrate $(Al_2O_3,3H_2O)$ obtained, which is equivalent to approximately 696,000 calories for each ton of leucite treated, assuming a production of 336 kg. of aluminium hydrate corresponding to 220 kg. of Al_2O_3. In addition, there is, however, the evaporation of the water associated with the HCl, which will absorb 10·8 calories per gramme molecule, so that about 149,000 additional calories are required for the vaporisation of the 233 kg. of water corresponding

to the ton of leucite treated ; thus a total of approximately 836,000 calories is required for the whole operation of converting the hydrated aluminium chloride obtained from the treatment of one ton of leucite into aluminium hydrate.

Taking factory operations, handling, say, 36 tons of leucite per day, about 5 tons of hydrochloric acid would be required, to which must be added any loss of hydrochloric acid which may be due to faulty chemical engineering. The cost of fuel for the treatment of this amount of material would be approximately 3s. per ton of leucite treated, and the products obtained would amount to 10¼ tons of potassium chloride, 8 tons of alumina, and 9 tons of silica. This estimate supposes a 93 per cent. yield.

I regret that I am unable to give views of the actual plant used to these processes, whilst the photographs in my possession of the small scale experimental plant in which the technical details were elaborated are too complicated to be of any value. The first factory experiments were performed in an ordinary stoneware tower, fitted with a plunger pump for the circulation of the liquid. The hydrochloric acid used was obtained by burning chlorine in hydrogen gas, produced from the electrolytic soda plant of the " Pomilio " factory at Naples. The commercial possibilities of this process depend mainly on the cost of the hydrochloric acid and the chemical engineering. In a factory where chlorine is a considerable by-product a careful consideration of costs will show the excellent commercial possibilities of this method of treatment of leucite.

The chemical-engineering problem of designing a plant for the manufacture of alum and silicate of soda presents no difficulty, since the ordinary materials of construction used in handling such products can be adopted, and apart from the provision of a circulating pump the plant may be designed in very many forms without serious effect on its efficiency. In the case, however, of hydrochloric acid treatment, on account of the volatility of the gas and the limited number of constructional materials available, the problem is far more difficult. If stoneware is used, the rise in temperature which occurs during the process may bring about stresses and failure through the serious cost in renewals and repairs. The removal of the silica obtained increases the difficulty of the problem if the usual methods are employed. Since the dissolving operation does not involve a temperature higher than that of boiling water, materials containing rubber may be used, and recently a new material containing a small amount of rubber has been produced, which seems to have all the qualities required for the construction of apparatus for this purpose. There is little doubt that soft rubber suitably prepared and supported by wood framing may also be used with success. In the plants erected on the Continent, stoneware, ebonite and soft rubber are used, but it would appear that soft rubber or a composition of rubber might be used entirely, provided that the problem of dissipation of heat is properly attended to. Silica, cerotherm and similar materials might be used for those parts of the apparatus where heat dissipation occurs,

and by the use of rubber connexions stresses could be avoided.

The removal of the silica formed can be readily accomplished by means of ejectors. This method avoids doors, joints and stresses, and at the same time the operation is more rapidly and more efficiently conducted. Compressed air or steam may be used as the motive fluid, but as a rule steam should be preferred. The material of which they should be made must be hard and acid-resisting. For general purposes there is nothing better than a silica ejector, the body being protected by a rubber casing. Fig. 5 is a drawing of a useful type. Soft rubber tubing is carried from leg C to the vessel to be emptied, steam is admitted by a flexible hose by leg A, and a rubber hose for delivery is connected to leg B. The granular mass associated with washing water in the dissolver is blown by the
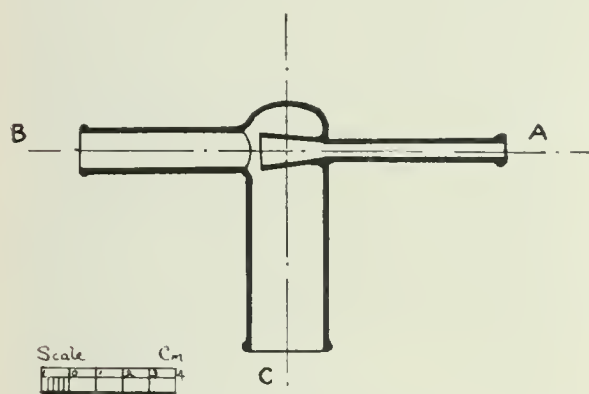
Silica Ejector. Sectional Elevation
Fig. 5

ejector on to a porous brick filter bed or draining bin, from which it may be taken for drying, air separation, etc.

These processes are due to Baron Blanc. Several other processes have been worked out, but have not been extensively used. The process of Dr. Hæge for making a phospho-potassic fertiliser has been worked to some extent. An acid process using nitric acid, an electric furnace process for the production of potassium carbonate and alumina, and a method of producing caustic potash call for some remark. The two last are due to Felix Jourdain. For producing caustic potash, powdered leucite and slaked lime with water are placed in an autoclave and treated for 3 hours at a pressure of 200 lb. per sq. in. The volume of the mass increases three times, and after two operations a solution containing 12 per cent. KOH is obtained. In a typical run with a 5-litre autoclave, 500 g. of powdered leucite, 400 g. of hydrated lime and 3 litres of water produced 51·6 g. of KOH, a yield of 95 per cent. Chemical engineering difficulties of handling the material, separating and concentrating the solution, are present, but the process is attractive. The residue in the autoclave is an excellent cement.

DISCUSSION.

Dr. W. R. Ormandy said Prof. Hinchley had stated that the lava contained leucite in crystals, which were both large and small, but in working them up to pieces of an average grain size of 1-16th in., he would like to know whether the bulk of the lava contained fine crystals or those which were near that size. As regards the manufacture of sodium silicate, he had been under the impression that the sodium silicates of commerce always contained more silica than corresponded to the formula $2NaHO + SiO_2$, given by Prof. Hinchley. Certainly the large amounts of silicates made in this country, when he was connected with the manufacture, were much more acid. Another important matter referred to by Prof. Hinchley was the manufacturing plant for dealing with hydrochloric acid. There was a material which had been developed in Switzerland, which had proved exceedingly useful in the conversion of wood cellulose into glucose, by means of warm, fuming hydrochloric acid, namely, Proderite, which would stand the action of concentrated, fuming hydrochloric acid or gas up to 60° C. Prof. Hinchley had also referred to the value of the process as a possible means of making alumina for the manufacture of aluminium. He had been told that the cost per ton of metallic aluminium for filter cloths in the manufacture of alumina by ordinary methods was £6, but even if that figure be not agreed, it was some measure of the enormous wear and tear that takes place on the filter cloths during this operation. He was exceedingly obliged to Prof. Hinchley for the information he had given with regard to the transformation of aluminium chloride ($12H_2O$) into Al_2O_3. If the people responsible for the operations in Italy could find a means for cheaply converting the hydrated aluminium chloride into anhydrous aluminium chloride, it would serve a tremendously important interest in regard to the refining and cracking of oils. If we could get cheap aluminium chloride in the presence or absence of iron—it was immaterial for this purpose—it would be a most valuable method for the conversion of heavy oils into petrols, for in spite of the fact that petrol is cheap to-day, the time is imminent when we might be called on for a method for the conversion of heavy oils into light oils.

Dr. J. A. Voelcker said that Prof. Hinchley had introduced to their notice a new material, for which he thought there was a future. Chemical manufacturers would not be able long to neglect it, for, not only did it provide potash, but also the possibilities of obtaining from it alumina and other products of material value. He would be forgiven if he did not wander out of his own particular subject, and if he dealt more particularly with the agricultural aspect. Everybody knows how great is the need for potash, how it is one of the chief wants of our soils, how much it is needed by all crops, and how we are always on the look-out for some fresh sources of supply. During the war the possibilities of potash starvation were prominently before us, but, with the war over, we have fresh sources open to us. None of them, however, provide such a cheap and valuable source as that referred to by Prof. Hinchley. For a long time the attempt had been

made to ascertain whether potash could be obtainable from cheap sources, such as granite and volcanic rocks, and agriculturists had tried whether any benefit could be obtained by using these, either very finely ground, or treated in some way or other. Hitherto, these attempts had failed. Although he had tried these materials for several years at his own experimental station at Woburn, practically no benefit had been obtained from them. There was, however, one exception, to which Prof. Hinchley had made reference, and that was the possible influence which silicates might have in regard to crops, and, from what he (the speaker) had ascertained in his own experiments, he believed there was considerable advantage to be gained in the use of certain silicates. In experimenting on different forms of lime, he had found, somewhat to his surprise, that, whilst the silicates in such materials as granite, phonolit, felspar, etc., were not available, he obtained distinctly beneficial results from the use of calcium silicate, and had reason to believe that the application of ordinary burnt lime to the land resulted in many cases in the partial formation of silicates of lime in the soil, a material which possessed distinct fertilising value. However, to come to the particular material which Prof. Hinchley had dealt with, when it was introduced to him he (the speaker) had recollections of other things of a similar nature and of other trials, and he could not say that he looked upon leucite very hopefully. But, on examining the material, he had found that a very large quantity of it was soluble in hydrochloric acid. He got something like 16 per cent. of potash soluble in hydrochloric acid, and, even with such a weak acid as citric acid—the ordinary 2 per cent. solution used in agriculture—there was about 1·8 per cent. of potash soluble. Therefore, he was inclined to place this material on a different footing from certain others which he had tried before. He had experimented with it, and was now in the third year of the work. Naturally, one looked to see what this material would possibly replace, and the most natural thing one would think of in comparison with it would be sulphate of potash, inasmuch as that salt has not the complications possessed by kainit, and sylvinit, and which bring in questions of the influence of magnesia, soda, etc., as well as of the potash. Accordingly, he compared sulphate of potash and leucite, and so arranged the experiments that, taking as the basis of application what one might consider a reasonable one, namely, 5 cwt. of leucite per acre, sulphate of potash supplying exactly the same amount of potash was employed. These experiments were tried in pot-culture work on wheat, and also in the field on mangolds and potatoes, and the results in the first year went to show that, on the soil he used—one deficient in potash—the application of both sulphate of potash and leucite gave decided increases of crop in the case of wheat. As between the two fertilisers, there was practically nothing to choose ; the difference was not more than what might be put down to experimental error. Similarly good results were obtained in the field with mangolds and potatoes. Here, again, the differences did not amount to anything material, but in both cases there was an advantage where sulphate of potash or leucite were used.

These experiments were continued in 1922, and the results were of a somewhat similar nature. Now, however, he had started a much wider series, and had enlisted the help of two or three well-known agriculturists who were prepared to carry out experiments on the field scale under his superintendence. Such experiments would carry conviction to the practical men. Inasmuch as for over two years he had found in his experiments a distinct benefit to accrue from the use of leucite, he believed there was a future for it from the point of view of a fertiliser, because, so far as he knew, there was nothing which could be supplied at so cheap a rate, whilst it was in a form available for the crop to take it up. These were the main points that he wished to speak upon, but he would like to ask one question. Prof. Hinchley had mentioned that the " cones " contained no potash. He would like to know whether that was because of the decomposition of the leucite and subsequent washing-out of the potash.

Dr. E. H. Tripp said he would like to ask two questions : first, what was the size of the particles of the comminuted leucite used in agriculture ; and the second, what was the price per unit at which the K_2O could now be supplied in London, compared with the prices of the K_2O from Alsace and Strassfurt ?

Prof. J. W. Hinchley, in reply to the discussion, said, with regard to Dr. Ormandy's remarks as to the size of the crystals, if Dr. Ormandy would look at the samples, he would find that the crystals are rather large, and that the very tiny crystals did not occur in the particular deposit in Italy to which he had referred.

Prof. Hinchley, continuing, said that with regard to sodium silicate, he had given the formula for a simple silicate, but possibly there were lots of sodium silicates. They all, however, could be formed in the same way by using different proportions. No heating was required, and the solution worked just the same as ordinary silicate.

With regard to Dr. Voelcker's remarks, the cones which he had referred to were andresite, and not leucite at all.

Prof. Hinchley, replying to Dr. Tripp, said that he had asked two pertinent questions. First, with regard to the size of the particles of powdered leucite, if they examined the bottle which he showed, which was a sample of the material used on the land, it would seem to be very fine indeed, and the fineness of leucite is really an indication of the rapidity by which it is assimilated by the plant. If we used a coarse potash, the benefit from it would probably extend over a number of years. That was a point which Dr. Voelcker would probably confirm, namely, that in the fine powdered form the potash is at once available, and its effect is apparent in the first year. Then, as to the price of the potash in this leucite. He had explained how it was produced, and how it could be supplied, but he was not going to discuss price at that moment, because he did not think it was necessary. He would say this, however, that the cost of the production of leucite for agricultural purposes is not half the cost of kainit or sylvinit.

The Chairman proposed a hearty vote of thanks to Prof. Hinchley, which was cordially responded to.

THE TREATMENT OF WATER FOR INDUSTRIAL PURPOSES.

A symposium on the above subject was held at a joint meeting of the Chemical Engineering Group of the Society of Chemical Industry and the Institution of Mechanical Engineers, in the Lecture Theatre of the Institution at Storey's Gate, Westminster, S.W. 1, on Tuesday, February 26, 1924.

Mr. W. H. PATCHELL (President of the Institution of Mechanical Engineers) presided, and was supported by Mr. J. ARTHUR REAVELL (Chairman of the Chemical Engineering Group).

The following papers were read :—

The Treatment of Condensing Water. By H. W. Coulson, M.B.E., A.M.Inst.C.E.

Water Purification for Industrial Purposes. By J. P. O'Callaghan, F.C.S.

Water Softening by Means of Doucil. By T. P. Hilditch and H. J. Wheaton.

General Methods of Water Purification for Industrial Purposes. By B. Heastie, B.Sc., A.M.Inst.C.E.

THE TREATMENT OF CONDENSING WATER.

By H. W. COULSON, M.B.E., A.M.Inst.C.E.

The need for a plentiful supply of water of suitable quality for industrial purposes has for long been well recognised, and many of the most important water using industries in the country are grouped around places where a naturally pure and soft water supply is available. There must, however, inevitably be cases where other factors determine the location of an industrial organisation, and the available water supply has to be utilised to the best advantage, and treated in such a manner as to be rendered suitable for the user's requirements.

The principles of filtration and softening of water supplies are well known, and can be economically applied to industries where the cost entailed by the

Hackney Condensing Data, 1922.

It will be noted that the vacuum periodically falls off badly with untreated water (see top L.H. of diagram). whilst the curve is much more even with the treated condensing water.

FIG. 1.

particular process bears a reasonable relation to the advantages which accrue from such treatment ; but in some cases the volume of water to be handled is so great and its use so incidental that considerations of purification along the usual lines are entirely outside the pale of economical possibility. A case in point is the treatment of the water used for circulating purposes in surface condensers, where a power station of moderate dimensions may circulate as much as one million gallons of water per hour.

rapid development of the organisms. This results in the formation of a gelatinous deposit upon the inner surfaces of the condenser tubes, and the deposit acts as an insulator to the passage of heat from the condensing steam.

As far as the author is aware, no attempt has ever been made until quite recently to treat economically large volumes of water which have immediately to be rejected after once passing through the circulating system, and on the face of it, it would appear as if no

FIG. 2.

FIG. 3.

Where the necessity of conserving the water supply entails the installation of cooling towers, water treatment plant can be, and is, economically applied to deal with the make-up, which may only amount to 5 per cent. or 10 per cent. of the total ; but this is only resorted to in cases where the water contains a large amount of encrusting salts, which would give trouble by the deposit of scale in the condensers.

The necessity for large quantities of water for cooling purposes frequently fixes the site of the power plant on the side of a river or canal. Nearly all such waters contain vegetable matter and micro-organisms, notably algæ, and the passage of the water through the plant raises it to a temperature which assists the

such process of treatment could ever be regarded as an economical proceeding. It has, however, recently been found that an exceedingly minute dose of chlorine added to the circulating water completely inhibits the formation of the jelly-like deposit which would otherwise act as an effective barrier to heat transference, leading to progressive loss of vacuum, and entailing frequent cleaning.

The maintenance of a high vacuum is a matter of supreme importance. It has been estimated that a variation of 1 in. affects the steam consumption—and therefore the fuel consumption—to the extent of about 6 per cent. It will be obvious that there is a very wide application for a method which maintains the condenser tubes always in a clean condition,

and therefore always at maximum efficiency, as compared with any arrangement whereby a progressive loss of vacuum eventually leads to a point where cleaning is imperative.

The quantity of chlorine required is extremely small—in most cases one-third of a part per million is sufficient to maintain the condenser tubes in a clean condition.

An objection is sometimes raised that corrosion of metal fittings might occur in the presence of chlorine. The experience gained at a number of power plants, some using as much as two million gallons of water per hour, where this treatment has been in use for the past two years shows that no corrosion whatever takes place. It would appear that the chlorine is immediately absorbed by the organic matter present in the water, as no free chlorine can be detected in samples collected from the condenser discharge.

The chart (Fig. 1) shows the effect of using untreated and chlorinated water upon the loss of vacuum, and has been prepared from data obtained by Mr. L. L. Robinson, M.Inst.C.E., to whom the author acknowledges his indebtedness. It clearly indicates the frequency of the cleanings which were necessary when using untreated water and the maintenance of a good vacuum after chlorination was introduced. It will be observed that these improved conditions continued in face of a rise in the temperature of the circulating water.

The drawing (Fig. 2) and photograph (Fig. 3) illustrate the Chloronome, an instrument which has

been developed for the chlorination of circulating and other waters. Chlorine is purchased in liquid form in steel cylinders, each of which contain about seventy pounds. The cylinders are connected to the Chloronome by suitable tubing.

When the cylinder valve is opened, the chlorine issues as gas and passes to the control board, which reduces it to a uniform low pressure, and measures the flow on a manometer, the rate being indicated by the level of the liquid in a glass U tube against a suitable scale. Stop and regulating valves are provided for controlling the flow of gas. On leaving the control board, the gas flows through an acid seal to the chlorine absorption tower, behind the board, where it is converted into an aqueous solution which is fed into the water requiring treatment. This method of dissolving the chlorine in a minor body of water facilitates the dissemination of the gas throughout the main body of water, and has a very important bearing upon the prevention of corrosion.

The complete apparatus occupies a very small space, and requires no attention other than a slight adjustment of the regulating valve to increase or reduce the flow of gas when more or less water is circulated, and the replacement of cylinders as the contents are exhausted.

The author thanks the Editor of the *Electrical Times* for permission to reproduce the chart in Fig. 1 which appeared in the issue of that paper on June 22, 1922, and the Paterson Engineering Co., Ltd., for the use of blocks of Figs. 2 and 3.

WATER PURIFICATION FOR INDUSTRIAL PURPOSES.

By J. P. O'CALLAGHAN, F.C.S.

The paper deals with its subject under the following heads :—

1. *Sedimentation and Filtration of Water for Works use, as applied to a turbid Water.* (Description of Beckton plant).
2. *Softening by the Lime-Soda Process.*
3. *Softening by the Zeolite (Permutit) Process.*

1.—SEDIMENTATION AND FILTRATION OF WATER.

Dealing first with purification from suspended matter, it is proposed to describe a combined sedimentation and filtration plant recently put into operation at the Beckton works of the Gas Light and Coke Company, capable of dealing with 4,000,000 gallons of Thames water per day. The river at Beckton is tidal and is frequently excessively turbid, so that a thoroughly efficient filtration plant is essential to prepare the water for works purposes. The plant consists of four large sedimentation basins and eight rapid sand filters of the gravity type and is particularly interesting on account of the high degree of efficiency obtained by the primary basins. The water to be treated is raised from the

river by centrifugal pumps driven by vertical spindle electric motors. The pumps are placed at about half-tide level, thus being entirely submerged at high tide, and having a maximum suction lift of approximately 14 ft. at extreme low tide. The delivery to the filtration plant is through two 12 in. e.i. mains. The filtration plant is built on 121 reinforced concrete piles 14 in. square and 38 ft. long, and comprises eight filters, housed in a suitable building, with adjoining basins for coagulation and sedimentation, together with a water tower for storage of cleansing water for washing the filter beds, air blower, pumping plant and filtered water reservoir, from which the supply is drawn and discharged into the works mains. Arrangements are included in the plant for the preparation of the necessary lime reagent used in the purification process and adding it to the water in suitable proportions. The water delivered to the plant from the river is received into a chamber fitted with baffling and diaphragm partitions, then passes into a measuring channel and overflows over two brass plate rectangular weirs.

For the chemical treatment, a definite proportion

of the total incoming flow is passed through the smaller weir into a Lassenhjort automatic measuring apparatus fitted with a chemical container, and the chemical valve of the latter is adjusted to deliver the exact quantity of lime to effect coagulation and precipitation of the suspended matter contained in crude Thames water.

The intimate admixture of the lime with the water is obtained by its fall into a trough and thence along distributing channels communicating with the coagulation and sedimentation tanks. These basins are of sufficient size to hold approximately 600,000 gallons of water, thus allowing for nearly four hours' time period for reaction and sedimentation when the plant is working at its full rated capacity. The design of the basins permits of speedy and efficient cleansing through the sludge valves and drain sewers. After allowing the heavier impurities and a considerable portion of the coagulated flocculent matter to be precipitated in the sedimentation basins the water is decanted over a trough communicating with the filter inlets.

The filter units, eight in number, are of the open gravity type. Each is 22 ft. long by 12 ft. wide, thus giving a total filtering area of 2,112 square feet. When the rated flow of 4,000,000 gallons per day is being dealt with and all the filters are working, each square foot of filtering area will pass 79 gallons of water per hour. The flow through the inlet valve of each filter passes along a system of communicating troughs placed above the surface of the sand bed, and the incoming water is thus distributed over the entire filtering area by overflowing the edges of these troughs. The filtering medium employed is a specially sifted and suitably graded mined quartz sand, supported on a bed of double-washed and graded gravel. Below this a manifold system of lateral collecting tubes fitted with safety strainers is arranged, which uniformly draws off the water from the entire filter-bed area. The filtered water thus collected is discharged through a central pipe and automatic rate of flow control valve into a chamber. From this it flows out through a weir into the filtered water channel, and thence to the reservoir sump of 60,000 gallons capacity, from whence it is picked up by high lift motor-driven centrifugal pumps and discharged to the works.

The filter beds are cleansed with air and wash-water. The operation is performed by agitating the filtering medium with air for about two minutes at a pressure of 3 to 5 lb. per square inch. A reverse current of cleansing water is then applied to the underside of the filter bed, and in its passage upwards through the bed, the impurities deposited on the sand are floated over into the collecting waste troughs and carried out through a waste pipe to the drain channel. In this manner each filter is thoroughly cleansed and restarted to work within the space of 10 to 12 minutes. The air for the aeration of filters is obtained from a motor-driven rotary blower. The filtered water used for washing the filters is raised from the filtered water reservoir to a tower storage reservoir having a capacity of 40,000 gallons and is drawn from there and used as required.

The water for use in the works, after treatment, is picked up by four sets of centrifugal pumps which have their suction in the reservoir or sump constructed directly underneath the pump room and these deliver into the company's main at a pressure of 80 lb. per square inch.

The coagulating and sedimentation tanks, filters, platforms, chemical house, inspection chambers, channelling, filtered-water sump and wash-water tower, are of reinforced concrete constructions. The floors of the pump room and the upper and lower platforms of the filter house are laid with terrazzo, and green glazed brick coping is laid around the filter tanks and platforms. The plant is one of the largest ever put to work and, by its use, the Gas Light and Coke Company relieve the London water supply of over a thousand million gallons per annum.

2.—SOFTENING BY THE LIME-SODA PROCESS.

The higher post-war cost price of fuel has forced boiler users to investigate more closely the efficiency of their steam-raising plant and, whilst as a rule the first step towards economy is taken by improving the stoking arrangements, it is now becoming more and more recognised that strict attention must be paid to softening the feed water if the fullest benefit is to be derived from coal-saving appliances. Steam-raising may be considered as a production into which two raw materials enter—viz., coal and water. These cannot be considered separately, but must be viewed as closely interdependent. The term " boiler evaporation " (meaning the number of lbs. of water turned into steam per lb. of coal burned) indicates this intimate relationship of fuel and feed.

The number of lbs. of coal burned or the number of lbs. of water evaporated taken singly are no indication of how economically or the reverse the boiler plant is working, but the ratio between them is the key to the whole question. A few figures will make this clear.

In a fairly economical boiler plant an evaporation of 8 lbs. of water for every pound of coal may be expected under favourable conditions. That means that for every 10,000 lbs., or 1000 gallons of water evaporated, 1250 lbs. of coal have been burnt. If the price of coal is 20s. per ton, it means that the cost of the coal required to evaporate 1000 gallons of water amounts to 12s.

If the feed water is hard (say, 20° Clark scale), about 3 lbs. of scale-forming matter is deposited in the boiler per 1000 gallons evaporated. The cost of softening such water may be estimated at about 6d. per 1000 gallons, including interest, depreciation, chemicals and attendance ; or, in other words, 4 per cent. of the coal cost. When it is considered that an economy of 10 to 15 per cent. may often be effected by simply softening the water, the economical argument for softening is obvious. But in addition to this, there are other great advantages that frequently escape the notice of boiler users. These are :—

(1.) The cleaning and chipping of the boilers can be done away with entirely, beyond going over the boiler once a year with a steel brush. This may and

often does mean a saving of more than £30 per annum per boiler.

(2.) The boiler does not need emptying nearly so frequently as would otherwise be the case. With coal at 20s. per ton the monetary equivalent of the heat loss every time a boiler is emptied and started up again may be taken for a Lancashire boiler at over £5.

(3.) The life of the boiler is increased, and repairs cut down to the minimum. A Lancashire boiler costs, nowadays, about £1200. If by treating the water the life of such a boiler is prolonged for even a few years, that alone handsomely repays the outlay on a water-softener. A large number of steam plants in this country would have a boiler to spare if proper treatment of the feed water was undertaken.

(4.) When economisers are in use their efficiency depends on the tubes being clean and free from heat-resisting scale. Great care is usually taken to keep the outside of the tubes scraped clean and free from deposit, whereas the inside (which is not seen) is often allowed to become almost solid with scale.

(5.) In the case of turbines, when hard water is used even as "make-up" to surface condensate, it is a frequent experience that the lime and magnesia salts are carried over in the steam and settle on the blades, thereby reducing the efficiency considerably. A properly operated water softener prevents this loss of efficiency.

These facts make it evident that the engineer in charge of a modern boiler installation cannot afford to neglect the water question. Pure water is more essential to success than good coal or good stoking, because without the former the latter are un-availing.

The following description of one of the modern plants may be of interest as typifying standard lime-soda apparatus as it exists to-day.

The outstanding feature of the lime-soda mixing and measuring apparatus is the device for the separate and exact measurement, in small, absolutely definite, and predetermined quantities, of both hard water and softening reagents. This device renders it possible to treat water or effluents of any character with any reagent, or combination of reagents, in rigidly proportionate quantities, which can be varied at will and adjusted to give any quantity desired over a wide range.

The water to be treated alternately fills each of the compartments of a two-chambered tipper oscillating on a shaft working in gunmetal-lined bearings. When one of these compartments is full of water the disturbance of equilibrium causes the tipper to over-balance and, by so doing, to discharge its contents of water into a tank; at the same time the other compartment of the tipper is brought under the orifice of the inlet pipe and filled in its turn with hard water, to be discharged in the same manner when full. In other words, the incoming water makes the tipper oscillate backwards and forwards, measuring off at each oscillation a definite quantity of hard water to be treated, and thus operating as a gravity water meter by means of which the quantity of water passing through the plant may be ascertained with absolute accuracy at any time by affixing a counter to the tipper shaft.

At each discharge of water from the tipper into the tank in which it works, a corresponding amount of water is displaced from this tank through a standpipe and chute into the reaction chamber of the softener, and here it receives at the same moment the requisite charge of chemical solution from the semicircular chemical container. This is effected by means of the positive chemical discharge valve placed in the bottom of the chemical container, which is opened at each movement of the tipper and caused to deliver into the reaction chamber the exact amount of softening reagent (in the majority of cases a mixture of lime and soda ash) required to soften the water to the guaranteed figure.

The lime-milk used has a strength of 10 per cent. : the lime-water used in earlier types of softeners had only an average strength of 0·13 per cent. ; the lime-milk, therefore, has a strength of nearly 100 times that of the lime-water, and produces a far quicker and more complete reaction. A further advantage of using lime-milk is that a known quantity of fresh lime can be mixed with a known quantity of water, a solution being obtained, the strength of which is definite.

POSITIVE VALVE FOR REAGENTS.

The part played by the patent positive chemical valve fitted in the chemical container to the plant is of such importance that a detailed description of this valve and its method of adjustment is desirable.

The valve consists of a fixed cylinder riveted to the bottom of the chemical reservoir into which screws an adjustable cylinder fixed in any desired position by a back-nut.

Within these cylinders work two valves, one of which screws on to a tail-piece projecting from the other.

The pitch of the threads on this tail-piece and the adjustable cylinder being the same, any movement of the cylinder results in a corresponding movement of the second valve, owing to this valve having a feather working in a key-way cut into the cylinder. The first valve is provided with a flat face and a piston body, which latter prevents any chemical solution being admitted into the adjustable cylinder until the lower valve has closed the outlet ports.

The operating gear consists of a double-lever fixed to the rocking shaft of the tipper. These levers are fixed to the vertical valve spindle by two loose links and trunnions clamped against a screwed sleeve by a lock-nut. These levers, when in operation, impart an up-and-down motion to the valve. The screwed sleeve works between rollers carried on to a bridge.

A weight keeps the valve tight on its seat.

RESULTS OBTAINED

Some results obtained by the Great Western Railway Company with a lime-soda plant similar to the one just described may be of interest. The water dealt with in this plant is derived from four different sources, the supplies from which are, however, mixed in constant proportion, giving a water the hardness of which, in the untreated state,

averages 17 to 18° Clark. The analysis of this water is as follows :—

				Grains per gall.
Silica..	..	..	..	0·17
Oxide of Iron	..	..	..	0·13
Sulphate of Lime ..	..	..	0·68
Carbonate of Lime	..	..	15·41
Nitrate of Calcium	..	..	1·79
Sulphate of Magnesia	..	..	0·90
Nitrate of Sodium..	..	..	2·63
Chloride of Sodium	..	..	1·64
Total Solids	..	..	..	23·35
Hardness	..	..	..	17·75°
Temporary Hardness	..	..	13·61°
Permanent Hardness	..	..	4·14°

The chemicals, determined by calculation and experiment, required to soften this water successfully are lime and soda in the following proportions :—

	Lb. per 1000 gall.
Lime (Best Fresh Burnt) ..	1·7
Soda ash (58 per cent. Pure Alkali)	0·5

A laboratory experiment with this water showed that it was possible by careful adjustment of the lime and soda to reduce the hardness to 1½° Clark. On the large scale the average hardness of the water after treatment was found to be 2° Clark, while the residual alkalinity (i.e., excess of lime and soda remaining in the water after the conclusion of the softening process) averaged only 1·5 grains per gallon, or 1½ parts in 70,000 parts by weight.

3. SOFTENING BY THE PERMUTIT ZEOLITE PROCESS.

The name " Zeolite " is applied to the hydrated silicates of alumina combined with sodium, potassium, calcium, magnesium and other metals. These substances possess the remarkable property of exchanging their metallic bases for others under appropriate conditions of contact and of effecting the exchange in the reverse direction. For instance, a zeolite containing sodium as its base is able to replace the sodium with calcium if exposed to contact with a solution of a calcium salt. When the calcium zeolite has been formed it can be readily reconverted into the sodium salt by similar treatment with a solution of sodium chloride. By the power of base-exchange which Permutit possesses, both calcium and magnesium are completely removed from water which is passed through a " Permutit " or zeolite softener. All traces of hardness disappear and the water when tested gives a ready lather with a minimum quantity of soap. The softeners are without moving parts, the entire process consisting of the simple filtration of the hard water through a bed of Permutit material. The installation of plant of the largest size, therefore, presents little difficulty, while in the case of the natural Permutit, lately adopted, depreciation is a practically negligible item, and the labour required to operate it is exceedingly small. As the softening power of a Permutit plant is drawn upon by the water itself in exact accordance with its hardness and its rate of flow, it follows that Permutit softening precludes the possibility of under-treatment or over-treatment. Variation in the quality and quantity of the water within the limits of the capacity of the plant makes no difference, whereas in such cases of variation a lime-soda water softener would require frequent readjustment.

The Permutit system works equally well whether operated under pressure or by gravity, and in consequence a softener can be fixed to the water service at any convenient level. This frequently saves double pumping, and allows of a practically unrestricted choice of position for the softening plant.

The base-exchange softening action continues until the active sodium in the Permutit molecule has been entirely replaced by calcium and magnesium. When this point has been reached regeneration is effected by passing a solution of sodium chloride slowly through the filter ; by this means the calcium and magnesium, as soluble chlorides, are expelled from the Permutit, their place being taken by sodium from the salt solution, thus reconstituting the original sodium Permutit, which is then as fresh and active as when first put to work. The alternating cycle of softening and regeneration is capable of indefinite prolongation.

Another type of Permutit has recently been introduced for industrial use. This new material, named " Permutit B," differs from ordinary Permutit both in physical characteristics and molecular constitution, these differences causing a radical change in the timing of the periodic cycle of softening and regeneration, enabling day and night continuous duty to be performed by one unit only instead of two. In the case of ordinary Permutit a single unit was usually designed to soften continuously for a maximum period of 10 or 12 hours, and it then required an equal length of time for regeneration. Therefore, day and night working involved employment of two units. In the case of Permutit B the plant is designed to operate for periods varying from three to six hours at a time, and is then completely exhausted. The material, however, is capable of extremely rapid regeneration, and a large industrial unit can be completely regenerated and recommissioned in an hour.

A most important advantage is that a reduced salt consumption in regeneration is possible with Permutit B as compared with the older material. Again, the new material is of a hardier physical character and, therefore, less susceptible to loss through wear and tear in general usage.

At several places combined lime-soda and Permutit systems have been installed, and are operating satisfactorily. It is not usually economical to treat a water entirely by the regenerative process where the hardness of the water is in excess of 40 grains per gallon, and in such cases a combined plant can be efficiently employed.

The Permutit process removes all the hardness produced by calcium and magnesium salts, and can, by a modification, be used to remove all iron, manganese and organic matter which the water may contain. It thus provides water which for all practical purposes is equal to distilled water.

A water, the hardness of which may vary frequently within wide limits, as some do, is taken care of automatically by Permutit, without any chemical control whatever. The usual daily analyses of the softened

water are unnecessary, leaving the chemist free to do other and more profitable work.

Wool washed with zero water, such as is produced by Permutit, is freed entirely from lime, while if washed with water of only 3° hardness, it will contain an appreciable amount. The impairment of wool by its lime contents and the difficulty it causes the dyer are only too well known. Experience shows that for each degree of hardness about $1\frac{1}{4}$ lb. of soap is destroyed per 1000 gallons of water treated.

The soap loss is the bugbear of manufacturers, and, moreover, is unavoidable, as the softening process completes itself with any soap that may be used before any benefit, such as is expected in the process of manufacture, can accrue.

It is not surprising that a Permutit plant installed in such a works should pay for itself in a short time by saving soap alone, without considering its other advantages. When soap plays the part of a water softener, insoluble calcium and magnesium soaps are formed, which are more or less irregularly deposited upon the material in process, whether silk, wool or cotton. This is also a constant source of trouble, causing irregular and uneven shades and difficulties in matching shades.

When silk is treated with soap in the process of manufacture, it is impossible to wash the soap completely from the skein on account of the formation of insoluble calcium and magnesium compounds. Moreover, in the subsequent weighting the tin does not adhere uniformly to the silk, owing to the lime and magnesium soap precipitates that have formed. The weighting is thus rendered very irregular. The disadvantages which result from the formation of these precipitates in the baths are well known to manufacturers and dyers. The important part which completely softened water plays in this branch of industry can therefore easily be realised.

In several manufacturing processes it is desirable that the water used should be absolutely free both from iron and manganese, and when these are present only in very minute quantities, it is almost impossible to remove them entirely by any of the older processes. A modification of the Permutit process makes this removal possible. By treating sodium Permutit with a manganese salt, a manganese Permutit is produced, and, upon passing water through layers of this arranged in an apparatus similar to that used to remove hardness, all the iron and manganese salts are oxidised and precipitated. This is not a true exchange action, but an oxidising process. At the same time, all organic matter and any bacteria which may be present are also oxidised, the manganese Permutit being periodically regenerated by the addition of solution of permanganate of potash. The process is very simple and thoroughly effective, costing for regeneration less than $\frac{1}{4}$d. per 1000 gallons.

The Permutit process is equally well adapted to the treatment of water for domestic use. Sulphate waters purified by the Permutit process are excellent for boiler plants. The whole of the scale-forming matter being removed from the water, there can be no scaling in the boiler and, as a consequence, it is unnecessary to open the boiler except for the annual inspection. All that is requisite is to see that the boilers are regularly blown off and occasionally emptied. Boilers fed with this water will show a marked increase in steaming capacity, reduced consumption of fuel and no cleaning costs.

WATER-SOFTENING BY MEANS OF DOUCIL.

By T. P. HILDITCH and H. J. WHEATON.

The softening of water by passage through a bed of base-exchanging material has become so familiar a process that it is only necessary to point out that lime and magnesia in hard water are exchanged for the equivalent of soda, and that the spent material is regenerated by passage of brine through it.

Hence the operating costs are the salt required, together with water (for solution of the salt and for washing the spent brine out of the filter and occasionally for flushing the filter) and the small amount of labour necessary to carry out this operation.

The chief characteristics of the softened water produced by this method are that it can be adjusted to any desired degree of hardness from complete softening or " zero hardness " upwards, and that it contains all the salts originally present in the water, except that salts of calcium and magnesium are converted into salts of sodium.

For different waters and purposes either the base exchange method or the lime-soda precipitation method may be the more suitable—the latter removes most of the dissolved calcium and magnesium carbonates and converts the other salts to the corresponding soda salts, but cannot reduce the hardness in practice to much below $2\frac{1}{2}$ parts of lime per 100,000 of water.

For many purposes it is held necessary to employ completely softened water, and even where this is not imperative the base-exchange process has frequently much to commend it in its simplicity of manipulation, whilst the cost of salt for regeneration compares very favourably nowadays with that of the chemicals used in the lime-soda process.

The present note is concerned with a description of a comparatively new base-exchange material manufactured by Messrs. J. Crosfield and Sons, Ltd., of Warrington, under the name of Doucil.

The oldest base-exchanging compounds on the market, the natural zeolites, consist of rounded granules of no great degree of porosity, and it is evident that all the softening action takes place very close to the external bounding surface of the grain.

Indeed, chemical analysis shows that but a small

proportion of the soda-content of a natural zeolite is available in water-softening.

Messrs. Crosfields' have been compelled to pay careful attention to the phenomena of surface action for a considerable period in quite other directions, and consideration of the experience thus gained indicated that if suitable porosity and surface could be secured it should be feasible to produce a base-exchanging compound in which much more of the combined soda could be utilised to produce soft water than in the cases of previous materials, natural or artificial.

This expectation was realised when it was discovered how to produce a complex silicate containing replaceable combined soda in the form of a dried gel.

The first type of doucil gel made (English Patent 142,974) was simply a very acid sodium silicate, made by the interaction of dilute solutions of sodium silicate and a mineral acid, or preferably of an acid salt, of which sodium bicarbonate is one of the most convenient to use.

These products, however, do not contain more than about 3 per cent. of combined sodium oxide, and not much more than one-third of this is available for base-exchange, the remainder being apparently converted into a calcium silicate.

The chemical interaction underlying the formation of the gel is rather interesting : with acid salts it is simply, of course, a case of withdrawal of soda from the silicate by the stronger acid, but it is curious to find that neutral salts of complex acids such as dichromates, pyroborates or metaphosphates show the same action, neutral salts of less complex acid being produced. With dichromates the action is easy to follow by the colour-change from orange to pale yellow, which occurs before gelatinisation sets in :—

For example,

$$(Na_2O(SiO_2)_3)_n + (n-1)Na_2Cr_2O_7 = \\ 2(n-1)Na_2CrO_4 + Na_2O(SiO_2)_{3n}.$$
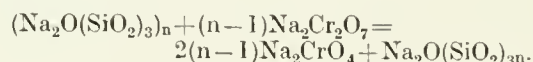
A similar process takes place when a dilute solution of ordinary sodium silicate, containing 2 to 4 molecules of silica to one of soda, is mixed with dilute sodium aluminate solution ; silica is a stronger acid than aluminium oxide, and sodium oxide is withdrawn from the aluminate with production of sodium metasilicate. The residual components enter into combination as a sodium alumino-silicate which is produced in the form of a gel (English Patent 177,746).

The final dried and washed product conforms closely to the anhydrous composition Na_2O, Al_2O_3, $(SiO_2)_5$, which demands 13·3% Na_2O, 22·0% Al_2O_3 and 64·7% SiO_2. Of the soda present, rather more than 3% is converted into non-exchangeable calcium silicate, and the remaining 10% can be completely replaced alternately by lime and by soda.

Of course, this complete replacement cannot be effected with simultaneous production of completely softened water, since this would require that the material should be as permeable to water as water itself, or that the granules should continue the base-exchange equilibrium internally as rapidly as the exterior surface action between the water and the parts of the doucil surface to which it has free access.

As a matter of fact an internal establishment of base-exchange equilibrium *does* proceed with the particles, but it is relatively slow compared with the exterior surface action. This is proved by the fact that a partially spent bed of doucil, if left for a few hours, is then able to soften water distinctly better than when the flow of water was previously interrupted.

For practical purposes, however, and especially if completely softened water is required, about 40% of the exchangeable soda, or 4% of the weight of anhydrous doucil present, is exchangeable for lime before the doucil allows appreciable quantities of calcium or magnesium salts to escape.

Doucil is marketed on a 50% moisture basis, so this is equivalent to saying that a ton of doucil will soften to " zero hardness " about 22,000 gallons of water, containing 20 parts of lime per 100,000 (or 25 grains of calcium carbonate per gallon).

(For base-exchange work it is convenient to base the hardness on lime rather than calcium carbonate ; also, with doucil, any magnesia present is calculated to its chemical equivalent of lime.)

To obtain a base-exchange capacity of this high order, two factors are necessary :—

(*a*) The doucil must be properly prepared.

(*b*) It must be used in a plant so designed that all the material is evenly exposed to the flow of water ; if the arrangement is such that the water can take a line of least resistance, plainly the doucil along that path will have to do all the work.

The correct preparation of the material is the responsibility of the manufacturer ; its correct use depends upon the firm which erects the containing plant.

It may be of interest to give a few particulars of each side of the question—production and application. In addition to maintaining the activity or base-exchanging capacity of doucil at its maximum it is desired to produce it in as hard a form as possible, and in grains of a size which combine maximum accessibility of water with as little resistance as possible to the flow of the latter.

The early stages of manufacture are all-important in securing both high activity and hardness of grain ; the generating solutions must be so mixed that the whole sets to a perfectly homogeneous mass of gel. This is controlled by careful adjustment of the concentrations of the aluminate and silicate solutions, and of their respective soda contents, and by suitable mixing devices. If clots of gelatinous precipitate or merely portions of different degrees of opacity (connoting variation in composition) appear in the gel mass, the hardness of the final product will suffer and in the factory such products, when accidentally encountered, are rejected.

The fresh gel is transferred without disturbance to a drier, in which it remains for four or five days whilst its water-content, originally about 90%, is reduced to about 50%. Too rapid drying, and especially drying at more than a very moderately elevated temperature, injures the final product. The dried gel is quite hard, and of a somewhat horny appearance, and is generally obtained by shrinkage during drying in the form of lumps about 2 in. in

diameter. These are washed in a slow stream of distilled water for some hours, during which the enmeshed sodium metasilicate, etc., is dissolved away, leaving the doucil in whitish lumps, which are submitted to further drying for about a day. This second drying assists to harden the material, and also renders it more easy to break down into grains of the desired size. The production of the latter is now a purely mechanical problem.

At first the manufacturers were unduly afraid of the resistance to water flow, and recommended the use of relatively large grains, at least in the lower part of the doucil bed. It was soon found, however, that softening capacity was being wasted in this way, and that the material was sufficiently hard not to pack down too tightly under water pressure when used in a smaller grade.

The preferred size is now from 1/8 to 1/20 in., whilst a still smaller size, 1/20 to 1/30 in., is sufficiently granular to be very useful as a top layer in a doucil filter.

A bed 6 ft. deep of the former size offers a resistance to waterflow of only about 1 lb. per sq. in. at normal rates of flow, whilst a 3 ft. bed of the smaller grade has a resistance of about 4 lb. per sq. in. under similar conditions.

Comparative measurements of resistance to disintegration by agitation under water have shown that the 1/8 to 1/20 in. grains of doucil lie between similar sized fragments of sandstone and of marble in this respect.

It may be remarked that the doucil is graded by passage through circular holes of the diameters given, and that in breaking down it splinters rather than crushes, so that the length of many of the doucil grains is greater than the upper limit of $\frac{1}{8}$ in.

Given suitable pipe arrangements for securing an even flow of water into and out of a doucil container over the whole of its cross-sectional area, no supporting bed of gravel or other inert material is necessary, and the doucil is simply charged into the container and, when the cover has been fitted, is washed upwards with water in order to settle the bed evenly.

It is worth repeating once again that it is of the utmost importance to ensure a perfectly even flow of water through the bed.

The depth of bed is largely a matter of convenience, but it is definitely proved that a deeper bed gives disproportionately better results, a 6 ft. bed, for example, softening about 15-20% more water than two 3 ft. beds of the same area.

Some indication has already been given of the water-softening capacity of doucil, and it only remains to be added that the capacity is naturally diminished if excessive amounts of sodium salts are present in the raw water, as these tend to reverse the base-exchange equilibrium.

The salt consumption is much reduced by using the last portion of the brine from one regeneration as the first half of the brine for the next regeneration (English Patent No. 203,497); in this way the consumption is about 8 lb. of salt per 1000 gallons of completely softened water of an original hardness of 20 parts of calcium oxide per 100,000. The amount of water used in solution of salt (5% brine) washing and occasional back-flushing does not exceed 4 or 5% of the water softened. The partially-spent and fresh brine should each occupy about 20 to 30 minutes in passage through the doucil bed, after which washing is carried out as rapidly as possible and need not exceed 15-20 minutes. The plant may be designed so as to be regenerated daily, weekly or at any suitable intervals.

The simplest way to operate a doucil plant is to pass the hard water downwards; regeneration should take place in the direction of upward flow, to minimise mixing of brine and water as far as possible, whilst displacement of the last portions of brine to a store-tank and subsequent washing are conducted in a downward direction.

A cubic foot of plant space contains about 35 lb. of doucil (50% moisture); thus to soften the 22,000 gallons of water previously referred to a plant capacity of about 64 cubic feet is necessary, e.g., a doucil bed 6 ft. deep in a cylindrical shell about 3 ft. 6 in. diameter by 8 ft. high would be suitable, with a brine tank capable of holding about 360 gallons of liquor.

Just as much as in the case of a catalyst, it is important to preserve the surface of the doucil grains in an absolutely clean condition, and therefore only water free from suspended matter should be admitted to the doucil bed. Suspended matter, especially if colloidal in nature, will tend to adhere to the active surface, and should be removed by prior passage through a mechanical filter. Similarly, any chemicals present in the water which might produce a precipitate at the surface of the doucil render such water unsuitable for treatment by doucil.

For all ordinary or average waters, however, the doucil method of softening is readily applicable and the following types of water-supply may be given as indications of its use :—

(i) Ordinary domestic supplies ;

(ii.) Supplies to laundries ;

(iii.) Supplies to dyeing, bleaching and other textile works (doucil-softened water is completely free from alkalinity after the material has been regenerated for the first time) ;

(iv.) Boiler feed water, at all events for steam-pressures up to 120 lb. per sq. in. (the base-exchange method is apt at present to give difficulties with corrosion on high-pressure boilers ; it is hoped later to devise methods which will eliminate this risk).

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GENERAL METHODS OF WATER PURIFICATION FOR INDUSTRIAL PURPOSES.

By B. HEASTIE, B.Sc., A.M.Inst.C.E.

Broadly speaking, the methods adopted for the purification of water may be divided into five classifications, which are as follows :—

(1) Distillation.

(2) Removal of suspended matter by filtration.

(3) Water purification by softening.

(4) Water softening by filtration through Zeolites.

(5) Use of boiler compounds.

Water which is required for the manufacture of industrial products must be such that the solids it contains do not modify to any appreciable degree the nature or the quality of the finished product. The conditions vary in each industry. However, absolutely pure water is very rarely necessary and it is generally sufficient to eliminate the deleterious elements. Thus, in the manufacture of starch, a water is required which is free from organic matters, ferments, ammonia and carbonic acid. Sugar houses require a water which contains very little salts so as not to increase the ash content of the sugar or brown the manufactured sugar. Breweries require a water which has to fulfil conditions determined for each kind of beer. These conditions have been particularly studied in Germany and generally result in waters freed from organic matters and toxic impurities, thus producing a soft water for the manufacture of brown beer, slightly chlorated and sulphated water for ale, hard water containing sulphated and alkaline earths for the manufacture of bitter beer.

For laundries a soft alkaline water is necessary. For dyers the water must naturally be clear and must not contain any salts which might act on the dyes. The manufacture of paper requires a water which is absolutely free from iron salts and preferably soft. The study of water treatment for each industry to give the best results is very long and complex, and manufacturers do not generally realise the enormous influence that the nature of the water used has on the cost of manufacture and the quality of the product.

The subject of most general interest is the treatment of water for boiler feed purposes. The tendency to-day is toward boilers of very large capacity, and high pressures, often working at 200 per cent. to 300 per cent. of their normal rating. The introduction of hard water into such boilers causes hard scaling on the whole of the heating surface in contact with the water. This scale, which is due particularly to alkaline salts, reduces the heat transfer, causing the flue gases to leave at a higher temperature than is desirable and involving a loss in thermal efficiency. Data illustrating this point will be given later.

It is evident, therefore, that one should reduce as completely as possible any solids that may be contained in the water.

PURIFICATION BY DISTILLATION

The ideal boiler feed is distilled water, as obtained by evaporation and condensation of natural water free from air. It is carried out on a large scale in electrical power stations, where one can see most clearly the deleterious effects of bad water feed and where efficiency is a first consideration—a small variation resulting in a profit or loss. Evaporators for the production of distilled water must fulfil certain conditions. The evaporation of raw water causes the formation of calcareous scaling and deposits which must be prevented from depositing upon the tubes. Various methods have been adopted with more or less success. High pressure evaporators usually scale up rapidly as a hard deposit is formed on the tubes due to the high temperature. To meet this difficulty the steam is passed through coils which can be easily removed for scaling. This is effected by passing cold water through the hot coils which contract and crack off the scale from their outer surface. In low pressure evaporators working at temperatures round 250° F. the water is passed through straight tubes, the scale is much softer and can be easily removed by means of a wire brush or scraper.

Evaporators working on the re-compression system, in which high-pressure steam is supplied to a steam ejector compressor, depend upon a very small temperature difference existing between the steam and water, about the order of 7° F., and thus the slightest deposit on the tubes is fatal. Any scale would increase the heat resistance, and temperature difference, and reduce the efficiency of the compressor, as is shown by the following figures :—

With steam at 200 lb./sq. in. gauge,

Vapour at 212° F. and 7·2° F. temperature difference,

1 lb. steam compresses 3·75 lb. vapour, giving 4·75 lb. of mixed steam at 219·2° F.

With vapour and steam in the same condition but 14·4° F., temperature difference, 1 lb. of steam only compresses 1·8 lb. of vapour.

One method of preventing scale is to introduce a quantity of sand into the tubes, the raw water being pumped through the tubes at a velocity of 8 to 12 ft. per second. Thus the tube surface is scoured and kept free from scale. The drawbacks of this scheme appear to be the power absorbed in driving the pumps, and the possible effect of sand on the pump bearings.

To obtain maximum thermal efficiency a distilling plant should be supplied with low-grade heat and operate with a small temperature drop.

Consider a generating station in which steam is supplied to a 10,000 kw. turbine at 260 lb. per sq. in. absolute, superheated 250° F. and exhausting to the condenser at 28·5 in. mercury. The total adiabatic heat drop is about 425 B.Th.U./lb. or assuming 77 per cent. thermal efficiency actually 326 B.Th.U./lb. The steam consumption on full load would be about 11 lb. per kw. hour, or 110,000 lb. Assuming 5 per cent. make-up water is required, 5,500 lb. of water must be distilled per hour.

Compare the two cases given below :—

1. A single effect evaporator using steam at boiler pressure 260 lb./sq. in., also superheated 250° F. The distilled water vapour passing to the condenser at 28½ in. vacuum.

2. A single effect evaporator using low-grade steam bled from the turbine at 21 in. vacuum (157° F.), the distilled water vapour passing to the condenser at 28½ in. vacuum (90° F.).

In order to make a fair comparison, assume that in both cases the condensed steam leaves the evaporator shell at 157° F.

Case I.—The available heat (per lb. of steam) is :— 1210 B.Th.U./lb. The latent heat of water vapour at 28½ in. vac. (90° F.) is 1041 B.Th.U./lb.

Therefore, neglecting radiation losses, 4,730 lb. steam are required to distil 5,500 lb. water.

Case II.—The total available heat per lb. steam is:— 1003 BTU. The latent heat of water vapour is 1041 BTU/lb. Therefore 5740 lb. of bled steam at 21 in. vac. are required.

Putting these steam consumptions in terms of energy :—

Case I.—4730 lb. steam, if utilised in the turbine would develop 430 kw. hours.

Case II.—The adiabatic heat drop over the turbine is 425 BTU/lb. Although in actual practice it is reduced by 20 per cent. to 30 per cent. due to unavoidable losses in passing through the turbine, the percentage loss in each stage (except the first one) is approximately equal.

Working under the steam conditions given, the theoretical drop between 21 in. and 28 in. vacuum is 98 BTU/lb.

Now, 11 lb. steam with a total drop of 425 BTU/lb. develops 1 kw. hr., therefore, $11 \times \frac{425}{98}$ or 47·7 lb. of steam expanding from 21 to 28½ in. vacuum gives out as energy 1 kw. hr. 5740 lb./hr. of bled steam are required for distillation.

This steam, if utilised in the turbine, would develop $\frac{5740}{47·7}$ or 120 kw. hours, thus a saving is effected of 310 kw. hours by using low-grade steam. Based upon a coal consumption of 1·8 lb. per kw. hour, the saving effected would be 6 tons per 24 hours, or assuming a load factor of 50 per cent., and coal at £1 per ton, the annual saving would be £1100.

In the above comparison it was assumed that the sensible heat of the high pressure steam was utilised, the condensed steam being cooled to 157° F. In practice the condensate would be discharged at approximately boiler pressure, a temperature of about 400° F. being utilised for heating. In this case the steam required would be increased to 6000 lb./hour (which if utilised in the turbine would generate 547 kw. hrs.), as the sensible heat would not be available. A common practice is to pass the boiler steam through a reducing valve supplying the evaporator at pressures of 70 to 100 lb./sq. in. : this does not affect the thermal aspect.

SUPERHEATED STEAM

The thermodynamic economy of evaporators can be greatly increased by operating in multiple effect. The number of effects which may be used is limited by the allowable temperature difference between the steam and water in each effect, also by capital cost and the ground space required.

The first question usually raised in discussing the merits of evaporating system is " How many pounds of water are distilled per pounds of steam ? "—It might be better put—" How many pounds of water are distilled per unit of energy contained in the steam ? " A comparison between three schemes is given in the following table, and again shows clearly the advantages of using low-grade heat for distillation.

ENERGY CONSUMED IN EVAPORATORS FOR PRODUCING DISTILLED WATER

Type of Evaporator	High Pressure Single Effect	Low Pressure Single Effect	Double Effect
Nature of steam supply to evaporator	Steam from boiler at 260 lb./sq. in. absolute. Superheated 250° F. Total temperature 650° F.	Bled steam from turbine at 4·7 lb./sq. in. absolute. 157° F. 21 in. vacuum	
Water distilled, lb. per hour	5500	5500	5500
Steam consumption, lb. per hour	4730	5740	2860
Available thermodynamic head expressed in kw. hrs.	430	120	60
Water distilled per kw.	12·8	45·8	91·7
Ratio :—Water distilled to steam supplied	1·16	0·96	1·92
Coal equivalent tons per annum	1430	398	199

Based upon 50 per cent. load factor and a coal consumption of 1·7 lb. per kw. hour.

In order to obtain equal thermo-dynamic efficiency in the high- and low-pressure single effect evaporators, the steam consumption in the former case must be decreased to 4·16 lb. of water distilled per lb. of steam, whilst to obtain a similar equality of efficiency with the double effect evaporators it is necessary for the steam consumption in a high-pressure single effect evaporator to give 8·3 lb. of distilled water per lb. of boiler steam.

In the above table, no allowance has been made for radiation losses ; however, these would naturally be much higher in the high-temperature plant, which would make the comparison more in favour of low pressure.

Water Purification by Chemical Means

When applied to boiler feed water, chemical methods of purification aim at removing from the water the calcareous salts which it contains, and which—decomposing in the boilers—cause scale to be deposited.

These salts can be divided into two categories : carbonates and non-carbonates.

1. In the first category belong almost all the salts in the most common waters ; bicarbonates of calcium and magnesium are the most important. In the usual process of purification, they are eliminated.

(a) By the addition of barytes or caustic lime, we have

$$Ca(HCO_3)_2 + CaO = 2CaCO_3 + H_2O.$$
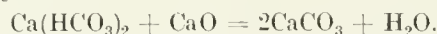
(b) By the addition of caustic soda, we have

$$Ca(HCO_3)_2 + 2NaOH = CaCO_3 + Na_2CO_3 + 2H_2O.$$
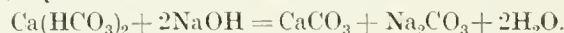
(c) By the addition of carbonate of soda (solvay soda)

$$Ca(HCO_3)_2 + Na_2CO_3 = CaCO_3 + 2NaHCO_3.$$
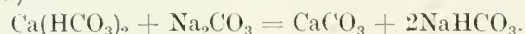
In all these cases the insoluble calcium or magnesium carbonate is precipitated, and the water fed to the boilers is always alkaline.

2. In the second category belong a great number of salts, of which sulphate of lime and chloride of magnesium may be mentioned. Chloride of sodium cannot be eliminated by practicable chemical methods from any water. Carbonate of soda or caustic soda removes sulphate of lime and chloride of magnesium ;

$$Na_2CO_3 + CaSO_4 = CaCO_3 + Na_2SO_4$$
$$MgCl_2 + Na_2CO_3 = MgCO_3 + NaCl.$$
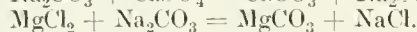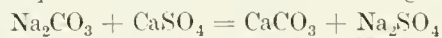
Thus one precipitates the $CaCO_3$, at the same time enriching the water in alkaline salts. During these changes, the reactions are not quantitative ; they give rise to a state of equilibrium, and to move this equilibrium in the favourable direction—that is to say, in the direction giving the desired precipitate— one is obliged to introduce an excess of the precipitating reagent, say carbonate of soda. The water introduced in the boiler is then, in all cases, alkaline and contains carbonate of soda.

Effect of Temperature on Chemical Methods

It is advisable to carry out purification at a high temperature, for the following reasons :—

(1) Gases in solution in the water are driven off by increasing temperature.

(2) Increased speed of reactions.

(3) Precipitation of the salts is much quicker at higher temperatures.

(4) Above 160° F. the bicarbonates of the alkaline earths begin to decompose, giving carbon dioxide and the carbonate.

(5) Diminution of the solubility of calcium sulphate. (10 per cent. diminution between 60° F. and 180° F.) and a consequent precipitation of part of this salt without any reagent.

Continuous Blowdown of Boilers

When a boiler, in which the water has been purified by some means, has been working for some time all the salts contained in the purified water are concentrated. These salts are alkaline salts only when the water is perfectly purified ; but are calcareous muds when purification is incomplete. Generally, there remains in solution in the water from 30—60 mgs. per litre of calcium carbonate. When concentration, produced by evaporation, becomes sufficient for precipitation of the carbonate in spite of the high temperature, it is deposited on the walls of the boiler and the tubes, forming incrustations, which become harder the slower the rate of deposition. To avoid this, the boiler is blown down, thus removing the mud collected. This water is at the pressure and temperature of the boiler and, in blowing down, a large amount of heat is lost, as in order to be effective, the blowing down should be of considerable volume in proportion to the total volume of the boiler. In the Kestner system, which is in extensive use on the Continent, instead of sending to waste a large quantity of water, a small amount is continuously blown down ; this cools, at the same time giving up heat to the feed water in the purifier. It loses on cooling a certain amount of the dissolved salts, and these muds are deposited in a settling vessel. It then mixes with the feed water and returns to the boiler. The removal of the muds deposited by the blowdown is carried out quite easily, as precipitation takes place in a vessel of conical form. When a boiler is blown down—e.g., using 20 per cent. of the feed water, its contents will be renewed every five hours ; this cannot be done by any other method which ensures elimination of the muds before they have time to accumulate. A constant salt content also is maintained in the boiler, and the amount of soda to be added to the water is greatly reduced.

Feeding with Purified Water

To explain the method of working which takes place in a closed circuit : suppose this already existing, and start at a given point, say, the boiler. There will be found a water rich in carbonate of soda. At the high temperature of the boiler, the carbonate of soda is partially decomposed, thus :—

$$Na_2CO_3 + H_2O = CO_2 + 2NaOH$$
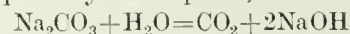
This decomposition increases as the temperature is raised, and the boiling prolonged ; thus the concentration of Na_2CO_3 decreases, due to the dissociation. While this is taking place, there is a concentration of calcareous salts which may have been entrained ; also a precipitation of carbonates and sulphate of lime, which are the commonest substances in boiler sludge. The blowing down of the water is carried out in the following way :—The blowdown is reduced by a special valve from boiler pressure to 15 lb. sq. in. It arrives at the ejector which condenses the flash-steam by means of the water to be purified. The raw

water, after the addition of a measured quantity of soda, is drawn in by the circulation produced by the ejector. Thus, inside the central cone, there is a great agitation of the muds, which are carried in the current until they are dropped by the deflector at the bottom of the purifier. The purified water rises with a decreasing speed in the annular space, traverses the filter and leaves the plant clear and hot. The regulation of the raw water and blowdown is carried out by ball-float valves.

If the amount of blowdown is calculated so that it contains at a given temperature the amount of soda necessary for the purification of the water entering the boiler at the same time, it is easily seen that it will never be necessary to add carbonate of soda for the purification of the temporary hardness of the water. There will be a closed circuit containing a certain amount of soda, which will remain constant.

The water, which leaves the purifier, contains no calcareous salts and is about 190° F., and contains as carbonate and bicarbonate of soda exactly the same amount of alkali as the blowdown used to prepare it, since there has been added to the raw water soda necessary for the decomposition of the non-carbonates. It enters the boiler, maintaining there a strictly constant salt content. In the boiler the bicarbonate of soda is changed to carbonate of soda by loss of CO_2, the carbonate being almost entirely dissociated to caustic soda, and we arrive at the initial point in the cycle. Thus, theoretically, it would be sufficient to add to the water only the quantity of solvay soda necessary for the precipitation of the non-carbonate calcareous salts; but, owing to the loss of liquid in the removal of the mud, this quantity is insufficient.

Advantages of Continuous Blowdown.

It is claimed that purification by continuous blowdown presents advantages from the chemical point of view, as well as from the thermal, which other systems of purification do not possess. From the chemical point of view, the appreciable advantage is a considerable economy in the amount of reagent. For a river water the following comparative figures for the purification of 1 ton of water by different processes may be cited :—

Process using soda	266·6 g. of solvay soda
Process using lime and soda ..	110·89 g. of lime
	16·7 g. of solvay soda
Process using baryta	366·43 g. of baryta
Permutit process	160·7 g. brine
Process using continuous blowdown	16·7 g. of solvay soda

In addition to this economy in purifying reagent, other advantages are claimed over the older methods of purification.

Zeolite Softeners.

Permutit Process.—This type consists of a filter, the bed of which is usually composed of a hydrated silicate of aluminium carrying a large amount of loosely bound and chemically active sodium. When water containing calcium and magnesium salts is passed through this bed of zeolite, the sodium is exchanged for the calcium and magnesium, and the water leaving contains only small amounts of the substances.

After a certain time all the available sodium in the zeolite has been given up in exchange for the calcium and magnesium. The zeolite can then be regenerated by soaking the filter bed in a strong solution of common salt. The calcium and magnesium which has been absorbed by the zeolite are exchanged for the sodium of the sodium chloride, forming calcium and magnesium chlorides.

After steeping for some time the brine is drawn off and the free salt rinsed out until the water runs sweet. The zeolite is then again in its effective state.

The advantages claimed by makers of this type are :—

It produces a water which contains a very small amount of calcium and magnesium salts.

It is simple in operation and does not require expert attention.

No chemicals are fed into the water, hence fluctuations in the hardness of the water are without effect.

The only reagent is common salt, which is usually easy to obtain.

As no settling tank is required, the space occupied is less than a lime and soda softener (cold process).

Among its disadvantages are :—

The amount of sodium salts is increased, and if used for boiler feed water may cause foaming or priming.

The capacity fluctuates with the fluctuation in the calcium and magnesium salts present.

(This process is fully described elsewhere in this Volume. See p. 58.)

Purification by Physical Means

Purely physical purification can be realised by the action of heat, by the action of vacuum and by electrolysis.

Thermal purification is the result of the action of high temperature on water, without vaporisation. It has the advantage of not introducing soluble salts into the water; but in order to be efficacious it is necessary that the action of the heat should be prolonged. In these conditions and when using a temperature higher than 158° F. one gets a decomposition of the alkaline-earth bicarbonates, and the precipitation of the neutral carbonates as a result. This operation is favoured by the presence of calcareous salts caused by preceding operations, by the agitation of the water due to boiling and the action of the deposits on the surface of the apparatus. Theoretically, it seems difficult to eliminate sulphates of lime by this process. The principal advantage of thermal purification is that the cost is greatly reduced as the heat supplied for the purification of the water goes to the boiler. This method of purification is at the present time used in a number of works.

" Purification by vacuum " is effected in certain processes of water de-aeration; it would not be economical if it were required only for this purpose. It is carried out as follows :—The water is discharged in the form of fine sprays into a space where there is a high vacuum. The water is then carried to boiling point in a manner which is nearly explosive, the vapour and the gas from the water being then eliminated by means of a vacuum pump. The water

which is not vaporised gathers in the bottom of the apparatus from which it is taken by an extraction pump. Due to the reduction of pressure, a dissociation of the bicarbonates takes place, and the carbonate of lime is precipitated and may be separated by filtration. This process is only of interest if one wishes to de-aerate the water by vacuum, as its action is limited to the elimination of the carbonates.

The use of an electric current for the purification of water has been proposed for some considerable time. In this process, insulated electrodes are suspended in the interior of the boiler, and are connected to the positive pole of the dynamo, the negative pole being connected to the boiler shell. The current used is of the order of 1 amp., at a pressure of 6—12 volts. The results of this process, which has not completely left the experimental stage, are very little known, as no actual data have been published.

ELIMINATION OF IRON

Certain waters contain large quantities of iron, particularly when they are taken from great depths and have not flowed above ground.

The iron is found in a state of iron combinations : carbonates, sulphates and sometimes acid salts.

When the purification of the water is carried out by one of the processes indicated above, the precipitation of the iron takes place during purification and under action of the alkaline lime, or soda reagents. It is therefore not necessary, when the water is purified, as above, to arrange for a special apparatus for eliminating the iron. Certain waters, however, are not suitable for industrial use, simply owing to the presence of iron ; for example, in dye works and for the manufacture of paper. It is then necessary to eliminate the iron without carrying out a complete purification of the water.

OXIDATION BY AIR

The elimination of iron from water may be done by a very simple method. The ferrous salts are transformed into ferric salts by the action of the oxygen in the air. By allowing the water to remain in contact with the air one precipitates the iron as "rust," but this oxidation is slow and incomplete. Plants for this work are designed to encourage an intimate contact of the water with the air, based on the fact that it is necessary to absorb by the water 1 c.c. of oxygen in order to transform 10 mgm. of ferrous iron into the ferric combination. Filtration is always necessary after the action of the air. This filtration, however, is very easy as the flaky nature of the precipitated oxidised compound lends itself very well to this operation, but the blocking of the filters is very rapid and they need cleaning very frequently.

Manganese in solution in water as manganese salts, but in smaller quantities than the iron, presents the same difficulties and is eliminated in the same way. The elimination of iron not only takes out the iron, but owing to the flow, the contact with the air, and the action of the contact surfaces, a part of the carbonic acid gas half combined is eliminated, which causes a diminution of the temporary hardness,

and at the same time causing a deposit of carbonates of lime and magnesium in the absorption towers. M. Paris in a recent article gives the following figures :—

	Raw water	After treatment and elimination of iron
Permanent hardness..	2·7	2
Temporary hardness..	12·2	8·3
	Mgm. per litre	Mgm. per litre
Lime ..	120	98
Magnesium ..	20	11

OIL EXTRACTION

Before water can be used for any industrial purpose it must be freed of any oil present. Water that has been in contact with mechanical parts of engines or exhaust steam from engines is polluted by a mixture of small quantities of lubricants. The oil is not only mixed with the water, but is also in the water in emulsion so that a simple decantation only enables one to eliminate the drops which float on the surface, and does not eliminate the oil in emulsion ; therefore the water remains opalescent.

The quantities to be dealt with are generally small, usually a few centigrammes of oil per litre of water. Sometimes there are larger quantities, but that is due to bad or excessive oiling, and can be remedied. But whatever quantity of oil is in the water, it is increased in the boiler owing to concentration and the oil adheres to the walls and diminishes considerably the coefficient of heat transmission. A layer of one fiftieth inch thick in a boiler working at 150 lb./sq. in. pressure will increase the wall temperature by 360° F.

When the water is slightly alkaline there is a formation of froth, and an entrainment of water. Finally, the oil mixed with the muds produces an insulating mixture which causes intense super heating and a decomposition of the oil and also forms an organic body which is corrosive and attacks the steel plate. The separation can be carried out either on the vapour which contains the oil before condensation, or on the water itself.

The separation of oil from the vapour is carried out in apparatus fitted with baffles on which the jet of vapour is broken—thus depositing its oil, or in apparatus where the vapour circulates at a great speed in a helicoidal space. The drops of oil are projected towards the outside by centrifugal force where they accumulate and are then extracted.

The separation of oil from water can be carried out either mechanically or chemically, or by electrolysis.

1. *Mechanical.*—Mechanical oil separation can be obtained by simple filtration through coke. This absorbs the oil and quickly becomes greasy. The dimensions of these filters are very large, and the attention to the filtering medium and its replacement is not an economic proposition. Thus, in one installation of three filters of 64 ft. dia. and 8 ft. 3 in. height, the coke has to be replaced every fortnight.

The French navy use sponge filters, which have the property of absorbing the oil. Such filters work very well but need a lot of attention. The sponges have to be frequently washed to be kept in a good state of absorption.

Finally, there are many apparatus which embody forced circulation of water between partitions and baffles and overflows for extracting the accumulated oil. These apparatus have no action except on drops of oil and do not eliminate oil emulsion.

2. *Chemical.*—Oil separation can be carried out chemically; this is hardly a correct term because the effect obtained in this case is in reality a physical effect. It is carried out by adding a mixture of an iron solution and aluminium to the water, followed by the addition of soda or carbonate of soda. One obtains by this means a gelatinous and flaky precipitate which carries with it all the oil in the water including that which is in a state of emulsion. This precipitate is easily filtered and the water is then free from oil but it is, on the other hand, enriched by a soluble salt caused through the reaction which has served to produce the oil separating precipitate.

A number of compounds are on the market, the composition of which is not stated but which act according to the above principles.

There are also products whose action is based on the well-known property possessed by clay for absorbing oil.

The trouble with this process is its length of time, the decantation of clay being very slow, and the separation by filtration very difficult because of the very rapid clogging up of the filter.

3. *Electrolysis.*—The most up-to-date process for oil extracting is the electrolytic oil separator. It is based on the property possessed by an electric current passing through water of destroying oil emulsion and accumulating this in the form of frothy flakes which can be separated mechanically. The electrolysis is generally carried out in wooden tanks and by means of closely connected iron electrodes. The conductivity of the condensed water being insufficient to enable the passage of the current, one is obliged to carry it to three degrees hydrometric scale by the addition of raw water. The conductivity of the water being higher when hot, it is more advantageous to carry out the oil separation at a fairly high temperature.

The viscosity of the water being lower when hot than when cold the power required to entrain the particles of oil is also lower under these conditions. The oil being entrained during the passage of the current, rapidly coats one of the electrodes. The cleaning of the electrode is carried out by periodically reversing the direction of the current. The oil then rises to the surface, accumulates, is evacuated by an over-flow, and the water which has been separated from the oil is carefully filtered.

The current required is valued at 0·15 to 0·20 KW per ton of water treated.

DISCUSSION.

Mr. T. H. ADAMS (Assistant to Sir Henry Fowler, L.M. & S. Railway, Derby) said that, with the exception of a warning in Messrs. Hilditch and Wheaton's paper on "Water Softening by Doucil" regarding the use of such softened water for high-pressure boilers, no mention had been made of the elimination of the corrosive tendencies of certain waters. The company with which he was connected had for many

years used on a large scale a water, derived from a tunnel, which had been found to have a marked corrosive action on boilers, particularly locomotive boilers. Analysis of the water, which was remarkably constant in composition, failed to reveal any reason for this action, the average composition being :—

				Grains per gall.
Calcium carbonate	..	..	..	10·50
Magnesium carbonate	..	..	..	3·03
Magnesium sulphate	..	..	..	·81
Sodium sulphate	..	..	..	9·87
Sodium chloride	..	..	..	2·56
Silica	..	..	..	..	·76
Water not expelled at 130° C., organic matter, etc	..	..	..	2·97
				30·50
Alkalinity (calculated as calcium carbonate)	..	..	..	14·10
Hardness ..	..	..	..	..	14·77 degrees.

The dissolved oxygen and carbon dioxide were not at all excessive, being approximately 0·66 and 0·52 grains per gallon respectively. The steel plates and the various steel stays and rods were continuously and regularly corroded to such an extent as to very materially affect the life of boilers using this water exclusively or largely, and the efforts made to eradicate this tendency had not met with complete success. The first attempt to deal with this water was by the Archbutt-Deeley intermittent softening plant, without the carbonating device, and a test extending over a period of about two years showed that no real improvement had been achieved. A further test was commenced with the idea of using a definite excess of lime above that amount which was required for ordinary softening—*i.e.*, to about 3 deg. hardness and 4 grains alkalinity per gallon. Unfortunately, circumstances demanded that this test should not be continued for as long as it was hoped to do. Nevertheless, when the boilers were examined they were found to be in much better condition than were similar boilers doing exactly parallel duty on the untreated water. For various reasons the matter had to be dropped at this time, and when again taken up at a later period a continuous water-softening plant was experimentally installed with the idea of repeating the tests with excess of lime. The makers of the plant, however, could see no reason why the corrosive tendency should not be eliminated by normal softening, and their request that the tests should be carried out on the lines laid down by them was acceded to, but with the result that at the end of another long trial (about two years) it was found that, although the boilers were, of course, cleaner and more free from scale, there was more corrosion of a localised character with the softened water as compared with the untreated water. Possibly the amount of metal which had been removed by the corrosive action was no more in one case than in the other, but it was localised, and therefore deeper and more serious. The experiments were in all cases carried out on four locomotives—*i.e.*, one tank and one tender engine for each kind of water, the engines used being of corresponding designs and classes and doing similar duties. He would be glad if any of the authors or members present could suggest any explanation for the abnormal tendency of the water

in question. If the cause were known it should not be difficult to effect a cure.

Another interesting case was furnished by the action of condensed water treated by a precipitation and filtration method for the elimination of oil, where the reagents used were equivalent solutions of aluminium sulphate and sodium carbonate. For its original purpose the plant was quite effective, but the treated water had proved corrosive, the action being, no doubt, due to the carbon dioxide set free by the reaction between the chemicals. At first the corrosion was confined to the metal (sheet iron) of which the filter shell was constructed, and to obviate this the interior was painted with a protective coating of bituminous solution. This simply transferred the trouble to the distributing trough in the top back drum of the water-tube boiler, and when this was corroded so far as to become ineffective for the purpose for which it was intended, and allowed the feed water to enter the boiler at one point only instead of along the whole length of the drum, the corrosion commenced on the boiler tubes at the upper ends of the back row with rather serious results. To cure this trouble he had suggested the insertion in the hot well of a number of iron or steel baffle plates, or preferably to fix a series of pieces of expanded metal in order to offer a large surface on which the corrosive tendency could expend itself on a material which could be easily and cheaply renewed. As there was no superfluous room in the boiler-house, this seemed at the moment to be the only remedy to suggest.

Mr. W. MACNAB said he believed he had had the privilege of starting the first lime-soda purification plants in this country, in about 1885 or 1886. At that time the method was simply that of mixing the water in a regular way with lime and caustic soda and separating impurities by sedimentation in suitable plant, and in that way uncommonly good results were obtained—as good, he believed, as are obtained at present by that method. But the difference which had struck him mostly as between the process then and now was the excellence of the devices used at present for measuring the quantities of reagents so that they should automatically follow any variation in the quantity of water. He had to admit that in the old days it was not so easy to cope with variation in the quantity of water ; where there was a steady supply being taken, however, it was quite easy to adjust the plant and very excellent results were obtained.

Lieut.-Colonel F. G. HILL (of the Dorr Co.) mentioned the work which his company has been doing recently in water-softening plants in America. This was in connexion with the first system of water-softening mentioned in Mr. O'Callaghan's paper— i.e., chemical precipitation and sedimentation, followed by filtration. By the introduction of a " Dorr " thickener, or clarifier, between the mixing chamber and the sedimentation basins, the following advantages had been obtained :—

Considerable reduction in size of the sedimentation basins. A quick return of the sludge from the clarifier to the mixer had resulted in saving of chemicals and better coagulation. Elimination of incrustation of sand grains of the rapid sand filter.

Miss SNOWDEN (representing Mr. W. B. Parker, of the British Thomson-Houston Co., Ltd., Rugby), dealing with the determination of hardness, said she had had experience in the analysis of water, and would like to know if it was the general practice of those present to determine hardness always by the use of soap solution, or whether it was determined by the method of acid titration. Again, in softening water by the lime-soda method, she had found that occasionally the results obtained from a laboratory test did not agree with the results obtained in practice, and would like to know the experience of the meeting ; in the first place, with regard to the most favourable temperature at which the reagents should be added —e.g., the lime and soda—to a water which had a good deal of both temporary and permanent hardness, say, about 20 parts of each— because that was the water she was dealing with ; also, she would like to know whether that temperature was very much below the temperature at which the calcium bicarbonate would naturally decompose and precipitate the calcium carbonate without the addition of soda or lime. Again, if there were time, she would like to know what was the method generally adopted for the estimation of the free CO_2 in water, as well as the bicarbonate and the total. What was the experience of the meeting as to the best methods of determining those three portions of the carbon dioxide in the water separately ?

Mr. R. STEWART referred to the softening of water by means of barium carbonate. It was generally admitted, he said, that for most industrial purposes water was effectively softened by means of lime and soda, or by natural or artificial zeolites, and those well-established methods were so perfect in their way that it might appear almost hypercritical to find any fault with either. The only criticism he would venture to make was that the purified water contained all the soda derived from the material used in the treatment. This might not be a matter of any great moment where the water was used for laundry or scouring purposes, or even for Lancashire and similar boilers, although with waters containing excessive quantities of calcium or magnesium sulphates the amount of sodium sulphate resulting from the treatment might have to be reckoned with. This was most emphatically the case in certain processes and in certain industries. For instance, in brewing, whilst it was customary to employ a water heavily charged—either naturally or artificially— with calcium sulphate in the production of pale ales, a soft water was preferred for stout, for which purpose, however, a very hard water softened by the addition of soda would not be admissible. Again, one desideratum of the sugar refiner was a soft water as free as possible from alkaline salts, and the same might be said in regard to feed water for locomotive and water-tube boilers. For these and various other purposes recourse might be had to barium carbonate for the removal of calcium and magnesium sulphates. It was usual to use lime because of its cheapness to remove the temporary hardness, the rôle of the barium carbonate being, of course, the precipitation of the sulphates. An exchange took place of the bases and acid radicles so that the precipitate formed consisted of barium sulphate and calcium carbonate

or magnesium carbonate. It would be possible to substitute barium hydrate for the lime in removing the temporary hardness, and as a matter of fact this was sometimes done. When barium hydrate was added to water containing calcium carbonate held in solution by free CO_2, a double carbonate of barium and calcium was precipitated, and the barium carbonate so formed served to remove the calcium and magnesium sulphate. Let us imagine a water containing, say, 13·6 grains of calcium sulphate per gallon and 10 grains of calcium carbonate were taken, such a water would require, under ideal conditions, exactly 31·5 grains per gallon of barium hydrate. It would be seen, therefore, that by the

procuring supplies of precipitated barium carbonate of a suitable quality. Ground mineral carbonate, Witherite, was tried in some of the plants, but was found to be quite ineffective. Consequently, the process suffered something of a set-back.. Latterly there had been a revival of interest in this process, and the erection of some large plants was then under consideration.

Mr. B. HEASTIE, dealing with Mr. Adams' query as to the corrosive action of water in boilers, said his company had had a good deal of experience in dealing with this, and, some time ago, had put on the market an apparatus in which manganese steel shavings were used; the water was passed through this apparatus,
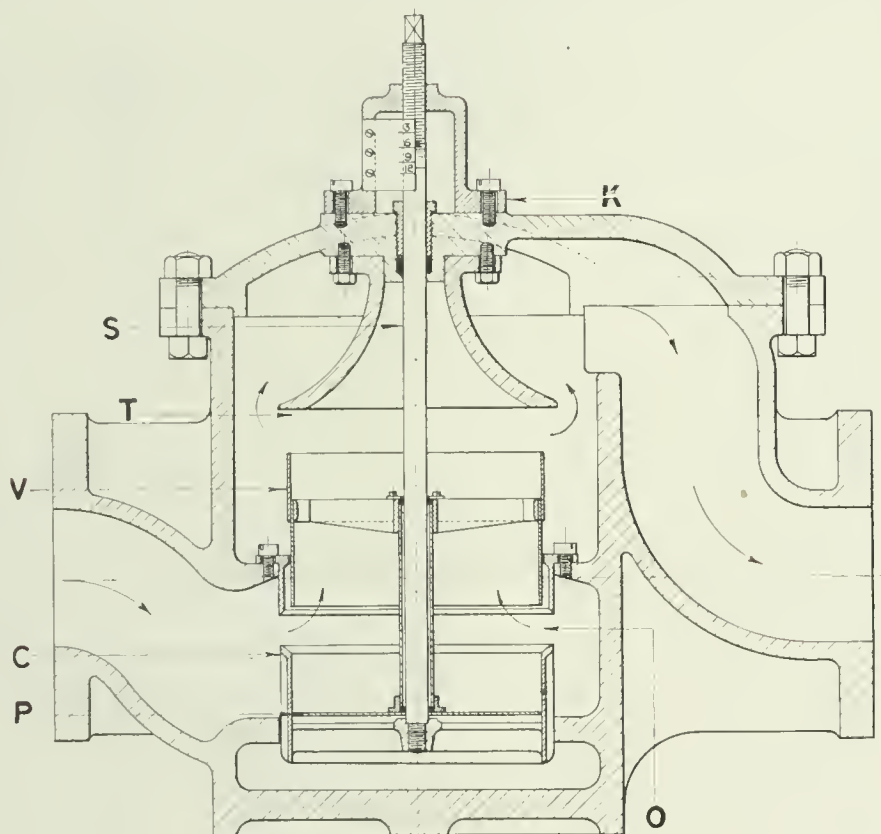
Fig. 1.

careful handling of the barium process it was possible to obtain a water almost free from everything (except, of course, any nitrates, chloride or alkaline bases present in the untreated water)—i.e., a water that for many purposes might replace distilled water. The barium process was introduced by Reisert, of Cologne, who obtained an English patent in 1902, and the process was described by the late Mr. John Royle in a paper read by him before the Manchester Section of the Society of Chemical Industry in 1906. Mr. Royle's firm (Messrs. Royles, Ltd., of Irlam) were the English agents for Reisert and the makers of the plant. He (Mr. Stewart) had been informed that the process had been well established in Germany. Several plants were erected in England prior to 1914, but during the war there was great difficulty in

with the result that the oxygen content of the water was reduced. The oxygen content of the average water was perhaps 5 or 6 c.c. per litre at normal temperatures, and the apparatus referred to had succeeded in reducing this down to less than 0·1 c.c. per litre. It had been found that in cases where boilers had corroded this apparatus had effected a complete cure, as in most cases corrosion had been traced to the oxygen in the water. He did not know whether Mr. Adams had taken careful tests of the oxygen content of the water he had used.

Mr. ADAMS said he had. There was about half a grain per gallon.

Mr. HEASTIE asked whether the boiler feed circuit was a closed circuit or an open circuit.

Mr. ADAMS said it was more or less an open circuit.

Mr. HEASTIE said that in some cases, even in closed circuits, they had been able to get down to about 2 c.c. per litre and there had been corrosion in the boilers.

Dr. NEWTON, at the request of Mr. O'Callaghan, replied to the points raised by Miss Snowden, and said he did so as a chemist engaged almost entirely in the analysis of water. The actual determination of hardness was made by calculation from the lime and magnesia found by analysis. Of course, soap would give the total hardness. Titration with acid would give the temporary hardness, or what was the same as the semi-combined lime in magnesium carbonate. With regard to the estimation of free carbonic acid, the method he had used for the last fifteen years was to take a measured quantity of water, admix barium and ammonium chloride and a measured quantity of barium hydrate, then allow it to stand over night in the laboratory and titrate the residual barium hydrate with acid the next morning. It if were desired that he should give details of this method he would be very pleased to do so.

Mr. J. ARTHUR REAVELL (Chairman of the Chemical Engineering Group), who proposed a vote of thanks to the authors of the papers, said that it was largely due to Mr. Patchell that these joint meetings took place originally. There had been a number of them, and they had all been very successful; the present one was another to add to the list. The more chemists and engineers got together the better for the whole industry of the country. That was the case, particularly in power stations, and there was no doubt that there was a big field there for the chemical engineer in connection with water treatment. In proposing a very hearty vote of thanks to those who had very kindly prepared the papers, he said that those papers contained a lot of matter which would be very useful and helpful in the future.

The vote of thanks was accorded with acclamation.

The CHAIRMAN, in closing the meeting, commented on the fact that boiler water had been the principal matter brought forward, and said that perhaps we did not realise that whereas we had been using boilers evaporating 3 lbs. per square foot per hour, we are now using them with three or four times that capacity. Therefore it was becoming more and more important to take very great care that not only clean but pure water was put into the boilers.

The meeting then closed.

Mr. J. L. HODGSON, M.I.Mech.E., wrote in connexion with Mr. O'Callaghan's paper on "Water Purification for Industrial Purposes" that it might be of interest to describe in brief detail the "Automatic Rate of Flow Control Valve" referred to in the third paragraph of the paper.

Some years ago the writer's attention was drawn to the fact that for efficient filtration it was necessary to have some simple device which could be set so as to deliver a given flow, and which when once set would continue to deliver this flow irrespective of the gradual increase in the resistance of the filter and of the inevitable variations in the "head" on the supply side of the device.

In order to meet this requirement the writer evolved the "Module," which is now in use at Beckton and elsewhere.

It consists essentially of a balanced throttle valve "V" moved vertically by a piston "P" on the same spindle, the piston being actuated by the difference of pressure across the controlling orifice "O." As soon as the flow for which the valve is set is slightly exceeded and the lifting force on the piston "P" due to the difference of pressure across the orifice "O" becomes greater than the weight of the piston and the balanced valve, this part rises and partially closes the throttling orifice "T"; thus throttling
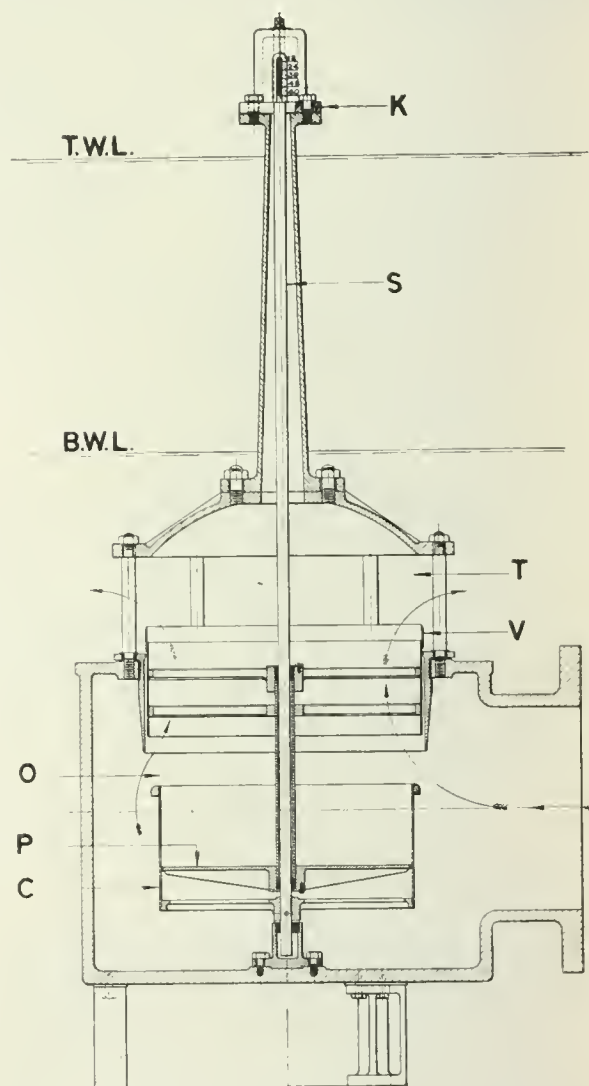
Fig. 2.

the flow until the load on the piston is again balanced by the drop of pressure across the controlling orifice "O." By this means a constant flow through the apparatus is maintained.

In order to set the flow to the desired amount, it is arranged that the piston chamber "C" can be raised or lowered by means of the central stalk "S"

so as to alter the width of the controlling orifice " O." A line engraved on the stalk shows, on a scale attached to top casting " K," the flow the Module is set to deal with.

The Module shown in Fig. 1 is similar to those used at Beckton. It is designed to maintain the flow constant at any value between 12,000 and 60,000 gallons per day. Owing to the salty nature of the water, the gunmetal parts, which are shown shaded darkly, were carefully insulated by means of vulcanite bushes and washers from the main body of the Module, which is made of cast iron. This procedure was necessary in order to prevent excessive corrosion of the iron parts.

The type of Module illustrated in Fig. 1 is also used for irrigation purposes, the body of the Module being then made of galvanised iron sheet

The Module shown in Fig. 2 is that adopted where it is essential that the whole of the Module shall work under pressure.

One of the most interesting recent applications of this particular form of Module is in controlling the rate of flow of air to the fermentation tanks in whisky distilleries, thus standardising the quality of the yeast produced and in consequence saving an enormous amount of time at bakeries owing to the fact that the standardised yeast will cause the dough to rise within a few minutes of a standard time. Yeast not so standardised is found to rise anywhere within half or three-quarters of an hour of the average time.

The balanced throttle valve shown in the figures is of some interest ; as until this valve was perfected the simple design of Module shown was impossible of realisation.

It will be seen that the valve consists of two concentric cylinders, and that the pressure on the two ends of the inner cylinder is always that of the liquid on the upstream side of the throttle valve ; while the pressure on the two ends of the outer cylinder is always that of the liquid on the downstream side. The upper edge of the outer cylinder is bevelled away as shown in order to prevent an unbalanced force being introduced by the suction which would otherwise act on this edge when the valve is nearly closed down.

CENTRIFUGAL DRYERS AND SEPARATORS— THEIR CONSTRUCTION AND USE

By E. A. ALLIOTT, Esq., B.Sc., A.M.I.Mech.E., M.I.Chem.E.

The arts of drying and separating by means of centrifugal machines are almost as widespread and varied as that of filtration. The action of the apparatus closely resembles that of gravity and one might, with justice, regard a centrifugal dryer as a draining board, and a centrifugal separator as a settling tank in which the force of gravity is replaced by a far greater one, arising out of the imposed acceleration. The unit centrifugal effect exerted by such means is most conveniently taken as the force in pounds exerted on a mass of 1 pound situated on the rim of the rotating basket.

It is proposed to deal with the subject in the following order :—

(1) *Centrifugal Dryers.*
    (a) Constructional Details.
    (b) Principles of Self-Balancing Machines.
    (c) Influence of Sundry Factors on the Material under Treatment.
    (d) Capacities, Labour and Output.
    (e) Special Machines.
(2) *Centrifugal Separators.*
    (a) Discussion of Features Presented by Emulsions and Suspensions.
    (b) Principles of Centrifugal Separation.
    (c) Practical Separators, with Sundry Data.

I.—CENTRIFUGAL DRYERS.

(a) CONSTRUCTIONAL DETAILS.

It will be best to begin with the basket which, of course, is the feature of the apparatus having the most important influence on the design. A type

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Fig. 1.

popular in the early days of centrifugals, and still in fairly considerable use for textile purposes, is constructed in wire, Fig. 1. These baskets were always supposed to give very free draining, although tests made by the writer show that it is not always easy to distinguish between the results obtained in such a basket and those from a plain perforated steel cage. In the latter case the ordinary perforations would range between $\frac{1}{8}$ in. in diameter for fine crystals and $\frac{3}{16}$ in. to $\frac{1}{4}$ in. diameter in the case of fabrics. Materials dealt with are often so fine grained that they would tend to pass through any ordinary perforations, and then it is necessary to line the machine in some way. This is nearly always done when the product is fed in the form of a slurry as even comparatively coarse crystals are liable to find their way through the perforations. In such cases, a fine-meshed gauze may be used, but this is perhaps less liable to become choked if it is not laid directly on the basket wall, but is supported by a more open netting. A cloth lining is sometimes employed and it is better to have a gauze of not too coarse mesh, to support it. The cloth lining must be properly selected and may be a strip laid on the periphery, and is then secured top and bottom by clamping pieces, or expanding rings. Long lengths of light cane laid over the cloth at the top and bottom have served the purpose quite well, as a temporary measure, and a fair joint is made, due to the centrifugal force exerted. In underdriven machines a cloth bag may be made to fit over the central dome and the outer edge is slipped over the basket rim. This is easily removable, and is suitable where food products or such substances as yeast, for instance, are being treated. Where linings are used, their life is apt to be shortened quite unnecessarily by carelessness in digging out the whizzed material. One way of avoiding this is to cover the surface, whether cloth or gauze, with a stronger netting of coarser mesh.

The materials of construction of the basket are fairly varied. Steel plate baskets are perhaps most widely used, and where they are not subjected to corrosion last for many years without repair. They are usually tinned or galvanised, and for some purposes are silvered. They are often coated with vulcanite, and sometimes with lead. Aluminium is used, and more seldom Monel metal or nickel. Steel baskets are made, having a porcelain inner cage, but this is a very special construction and examples are rarely seen. Wicker linings are not uncommon for some purposes.

One of the most important features of the basket is the method of discharge. In the case of small machines, or even large ones if isolated, it is often quite satisfactory to have the ordinary type of basket and empty over the side with a spud or shovel. For large plants and where capacity on a single product

is of first importance other methods have to be adopted. Textile products are the most important exceptions to this rule.

The ordinary type of basket may be fitted with two or more doors in the bottom, which may be opened at the conclusion of the operation, and the dried material pushed through an opening in the casing on to a shoot, which takes it clear of the machine.
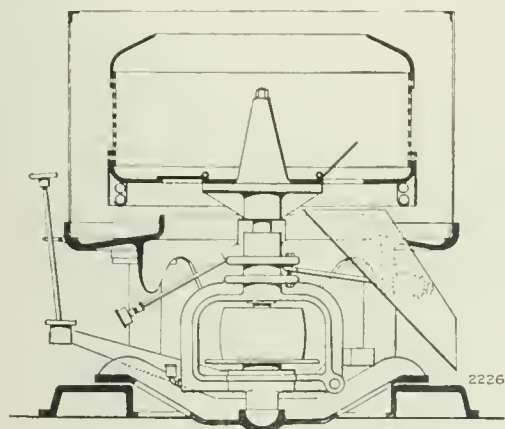

FIG. 2.

Fig. 2. Another method is to have a lifting basket, fitted with trunnions, over which shackles carried by a travelling pulley block may be slipped to lift the basket.

It may then be run along an overhead rail to a convenient point, and its contents tipped out, which may be very effectively assisted by the use of a lead mallet to jar the basket. This is extremely helpful for handling such awkward materials as metal turnings and so forth.

A variant on this scheme is to fit the basket with a loose upper ring and inner removable lining made in strong wire netting. The top ring is unclipped at the conclusion of the operation and the wire netting lifted out by means of a jib crane, which is built as a portion of the machine in question. This type has found particular favour for dealing with meat and fish offal.

Centre discharge baskets are widely used, and are standard in the sugar refining industry. Here the bottom of the basket has a large opening in the centre, containing a spider by means of which the basket is connected to its spindle. The opening is covered by a light conical valve.

This valve, when lifted, can be hung from a ring mounted on the shaft at a convenient height, or from the frame of the centrifugal. It is now an easy matter to push the materials between the spider arms, and if the floor of the basket is sloped, discharge in some cases is considerably facilitated. There is, however, another side to the question of sloping the basket bottom, for the effective capacity is thus diminished in proportion to its total height and there is less filter area per unit volume of basket content. If the product flows readily after spinning, there may be a net advantage, but if the material filters with difficulty, and is not easy to break down, this will be more doubtful.

One firm use an interesting device to save labour, in the case of a product which forms a very firm wall, which is difficult to break down. A length of chain is laid round the bottom corner of the basket. On pulling this out, the bottom of the wall is cut away, and it becomes easier to deal with.

A discharge scoop is sometimes employed where materials are suitable. One design consists of a specially shaped blade, which is mounted on a vertical shaft which can be worked up and down by means of a rack and handwheel. This is carried on an arm bolted to the top of the casing, and can be swung into position when required. The basket is run comparatively slowly during the operation, which is very rapidly accomplished. The writer was informed by one American refinery that the adoption of this device had halved the labour on the refined sugar centrifugals. He also saw it applied to sulphate of ammonia. It will need some caution in applying to other uses, where the wall of spun material might break away less readily.

Finally, for materials which fall away very freely, after drying, one can use a basket of the self-discharge type. This is unusually deep and the opening at the bottom is exceptionally wide, and is not covered by a valve.

Instead of this, a cone is fitted to the shaft about the centre of the basket, into which the incoming material is fed as the basket rotates. It is thus thrown outwards to the basket wall, where it adheres by centrifugal force and nothing can fall through the bottom opening until the machine stops. Then, in the case of a really loose material, which has not been subjected to excessive drying, in the machine—raw sugar for instance—the greater part will slip down of its own accord as soon as the machine stops. A little, however, will still remain and must be helped out by hand. Where the material tends to adhere and must be dried thoroughly, such a machine will not work with the same degree of success.

It will be further observed that the material must be fed into the machine in such a state that it will spread itself evenly over the basket wall, and the basket must be run sufficiently slowly during filling to allow a proper opportunity for this to occur before the slurry is made immobile by loss of liquor. This type of basket is therefore somewhat limited in application.

The speeds at which centrifugal baskets can be run is of great interest. The usual formula for centrifugal force in lbs. exerted by a mass of 1 lb. is

$$C = \frac{D\omega^2}{2g} = \frac{DN^2}{5866}$$

Where ω is the speed in radians per sec., N in revs. per minute, and D the diameter in feet. The above formula, however, can be expressed in another way, involving V, the surface, or peripheral speed, in feet per minute

$$C = \frac{V^2}{57,888D}$$

It will be seen from the latter expression, that the force exerted for a given surface speed must be inversely as the diameter. Now, a centrifugal

machine is very like a rotating pulley so far as its strength is concerned, and if the liquid load is proportional to the area of the basket wall, and to its thickness, bursting will always occur at the same surface speed, whatever the size of basket and whatever the thickness of wall. Surface speed is, therefore, the limiting factor so far as strength is concerned, and cannot be varied very much by the engineer, although he may do so by disregarding the question of proportionate loading. He cannot do a great deal by increasing the thickness of the basket wall, because the self-stress in the wall due to its own weight is a very considerable element and increases as the

per minute having a centrifugal effect of 512 approximately and a surface speed of 9424 ft. per min. If this 36 in. basket were of steel, the tensile self stress in the wall would be about 3000 lb. per square inch, depending on the perforations. If the load formed a 4 in. liquid layer of specific gravity 1, it would then exert a bursting pressure of about 65 lb. per square inch. These figures would, of course, vary a little for other sizes at the speeds on the chart.

It will be seen from the figure that though the surface speed increases in the case of the larger machines, the centrifugal force decreases. Ordinary
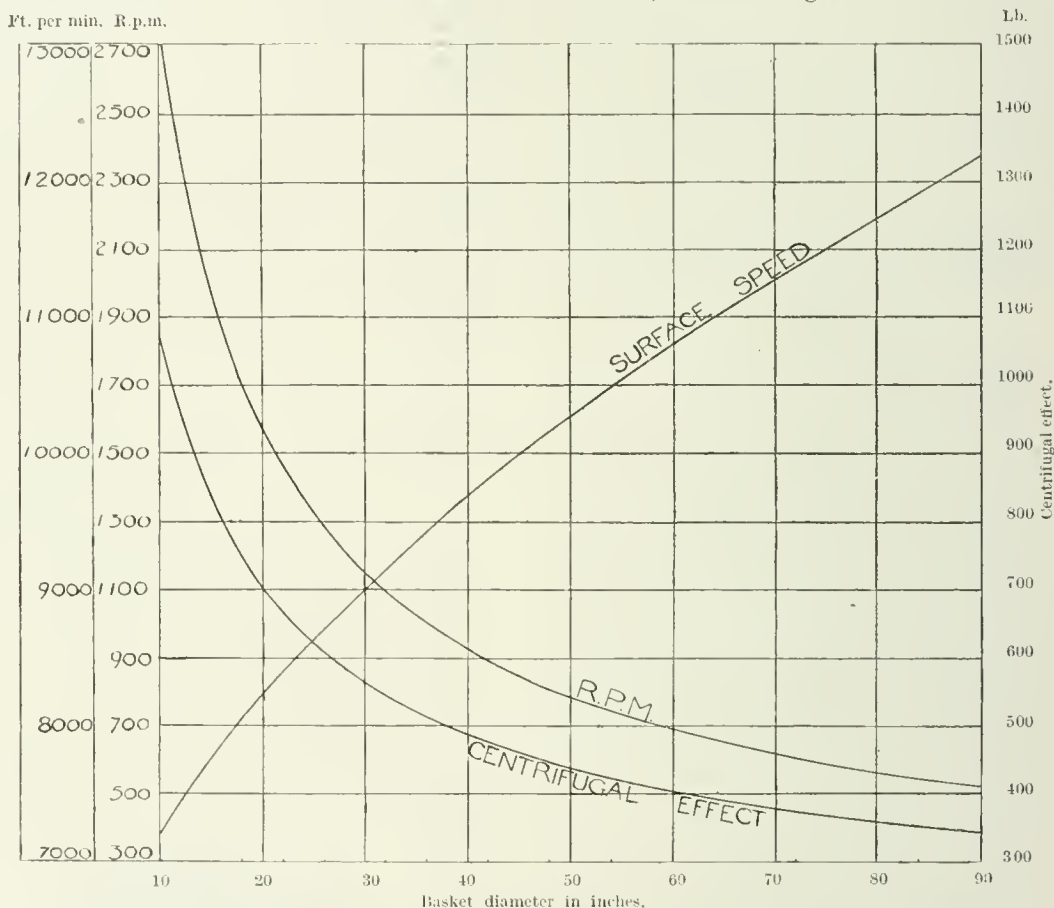
Chart showing relations between size, speed and effect of centrifugal baskets, as ordinarily based on a 36 in. basket at 1000 r.p.m.

FIG. 3.

square of the surface speed so that wide variations are rarely economical. Hence speeds usually vary between 8000 to 12,000 ft. per minute, though some smaller machines, 12 in. dia. and under, when used as separators, go as high as 16,000, or even 18,000 ft. per minute.

If the surface speed were kept constant, the formulae show us that the centrifugal effect would always be inversely proportional to the diameter, but in practice the running speed is often an average between that for equal surface speed and that for equal effect. Fig. 3 shows what the speeds of sundry sizes of basket would need to be if they were based in this manner on a 36 in. basket running at 1000 revs.

commercial speeds do not vary very widely as a rule from those shown, but in practice the smooth curve is apt to be replaced by a series of steps, which will vary for each maker.

When desirable, it is possible to use higher speeds than those shown on the chart. This, however, is seldom necessary, and requires special consideration in each case. It is preferable to confine any speeding up to cases where the load is of light specific gravity, not appreciably above 1, or of such a kind that it cannot exceed the net capacity of the basket. It must also be free from any tendency to build up unevenly.

The rules for the stress are not difficult, and it is

easily reckoned by anyone who can calculate the stress in a boiler. The centrifugal force of the rim itself must be considered, which gives the self-stress referred to above, and then there is that exerted by the load, which is usually estimated as if the whole were concentrated at the full diameter of the rim. This supposition is perfectly accurate for liquid loads, since a portion near to the centre of the basket makes up in hydraulic effects what it lacks in centrifugal force, for pressure applied to a small surface near the centre is transmuted into a proportionately larger total force at the full circumference of the rim.

For solid loads, the actual force will be somewhat smaller, though not very appreciably so unless the wall of the material is very thick. In any case, however, the error will be on the right side, because the unavoidable irregularities in the loading of solid materials impose strains on the basket that cannot be covered by any nice formula. Textiles are again an exception, for the fabric apparently takes up a large portion of the stress within its own structure. This is evidenced by the fact that baskets are often returned for repairs after working for long periods, and are then found to be in such a state that by ordinary rules they should have given out long before. The only guide here is experience, which will indicate what factor of safety may be properly used, and it ought always to be an ample one.

As compared with a boiler, a centrifugal basket has an advantage in that it is constantly under the eye of the operator, and any flaw or defect is likely to be observed. On the other hand, except when dealing with liquid loads and substances which form a very smooth and even wall, it is subject to all kinds of unequal stresses which are unknown in boiler design.

No very special features are presented by the pan or outer casing. It is usual to construct this in cast iron, in the case of "fixed" spindle machines. Steel is better for self balancing types, where there is even the remotest possibility that the revolving basket might strike the pan. Neither material should be looked on as a safeguard against the basket bursting. The only safeguards are to see that there is no possibility of running at excessive speeds, and maintain the basket in sound condition.

The outer casing may be lined with vulcanite, brass, aluminium or other materials.

When food-stuffs are being handled, such as yeast, patent preparations and so forth, it is desirable that not merely the interior of the basket, but the whole of the basket and the interior of the pan, shall be accessible for thorough cleaning. Openings may then be arranged in the side of the pan and provided with removable doors.

It may be interesting to look at the various methods of driving centrifugal machines. Perhaps one of the most common ways is by belt. Where belts are used in chemical works they are apt to be subject to rapid deterioration through fumes or through accidental splashing with the liquors being treated. It is therefore worth while to take some steps to protect them by fitting splash guards.

Steam-drive is useful as a matter of convenience.

In the case of under-driven fixed-spindle machines the engine may be bolted on to the pan and the drive transmitted to a crank formed in the spindles. Such machines are necessarily of the suspended type, since it is almost impossible to balance perfectly such small high-speed engines, but are very compact and convenient. The lubrication, however, needs careful attention, and the engine is subject to a good deal of wear and tear. A separate engine is much more satisfactory and special ones are made with horizontal pulleys for direct belt-drive for under-driven machines. On Weston machines a vertical engine is used mounted inside the swan neck or at one side of the gallows.

A turbine wheel may be fixed to the basket, but the steam consumption is high, and the arrangement is uneconomical unless it is required to steam the material in the basket, and the exhaust can be utilised for this purpose.

Electric drive is extremely clean and convenient and lends itself to various safety-devices. The most compact arrangement is to mount the motor with its spindle in line with that of the machine and to drive through a friction clutch.

Water-drive is convenient and economical where large batteries of machines have to be run. It is not, however, suitable for odd machines, as it requires a special steam-driven pump to circulate the water at the necessary pressure. Further, unless this pump is kept in proper order, the speed and efficiency of the centrifugals is apt to fall off. The driving mechanism is a Pelton wheel mounted on or in line with the spindle of the machine. There are two jets, both of which are in use for acceleration, but one automatically shuts off when full speed is attained. A governor may be used, but in many cases shutting off occurs through the waste water entering a specially arranged opening when the speed becomes sufficiently high and there raising a float which operates the trip mechanism.

In this connexion we have to mention friction clutches. Their use in the case of electrically driven machines has already been mentioned. It is necessary in order that excessive current shall not be taken when starting, and permits the satisfactory use of types of motor having a low starting torque and high initial power factor. Without its aid electric drive for centrifugals would be difficult except under direct current conditions, and large and costly motors would be requisite.

Such clutches are not necessary on steam or water driven types but are an advantage with all belt driven machines and a necessity if the latter are of large size. The time usually allowed for bringing the basket up to speed is about two minutes. Very fast acceleration cannot be obtained without annulling the chief advantages of the friction clutch, and serves very little purpose, as liquor is draining away nearly the whole of the acceleration period. Unduly slow acceleration, however, causes waste of power and is liable to give trouble in self-balancing machines, as will be seen.

Machines are very often fitted with guards. These are usually of the plain hinged type though various automatic ones have been devised. The simplest of

the latter consists of a loose disc which flies out eccentrically from the spindle as soon as this attains a little speed, and prevents the guard being lifted. This disc cannot be replaced until the machine has nearly come to a stop.

We are now in a position to look into the question of bearings. In the old types of machine, ball bearings were rarely used, and the spindle ran in bushes, which were usually made of phosphor bronze, and the thrust bearings or toe-step would often consist of several layers of brass and steel discs. The practice nowadays has turned more definitely towards ball, thrust and roller journal bearings. These can be run for long periods without any attention at all, but it is preferable that they should receive grease every six or eight months.

We may classify centrifugals into the following varieties :—

1. Fixed spindle machines.
2. Suspended machines.
3. " Free spindle " or self-balancing machines.

The latter class may be subdivided into the " peg-top " or underdriven and " Weston " or top-driven varieties.

In the fixed-spindle machines no other spindle motion is possible but that of rotation. The whole unbalanced centrifugal force is transmitted to the bearings, which must be amply proportioned to stand the strain. Bearing wear and tear is not at all excessive, in spite of frequent bad loading, and is generally negligible. Machines of this class often show some advantage in price, and may further be worked, in many cases, by the most unintelligent class of labour that is available.

They are not suitable, however, for mounting on upper floors, since any out-of-balance load has to be taken up ultimately by the foundations, and a period might be set up in the surrounding structure if it is not perfectly rigid. Further, vibration is sometimes transmitted for some distance, even when centrifugals are mounted on heavy concrete foundations in firm ground. This may be easily avoided by the use of suspended machines, which are ordinary fixed-spindle centrifugals hung by suspension rods from columns carried on an independent base-ring. These rods have hemispherical ends fitting in cups at top and bottom and the machine is therefore free to vibrate without affecting its surroundings. Such vibration is not, as a rule, large and even a really badly loaded machine will appear comparatively steady when once a high speed has been attained.

In the free spindle or self-balancing type of machine, the spindle bearings are mounted in a box or casing, to which a certain amount of free play is given, under sufficient constraint from rubber buffers or an arrangement of springs. The original reason for this method of construction was to allow bad loading to be taken up without imposing undue stress on the bearings which, in this instance, is limited by the elastic resistance of the supports. Perhaps the most important advantage gained in practice is the convenient arrangement of bottom discharge that is usual in top-driven machines of this class. Self-balancing machines are absolutely necessary where speeds are very high.

The most familiar of the various forms in use will now be described.
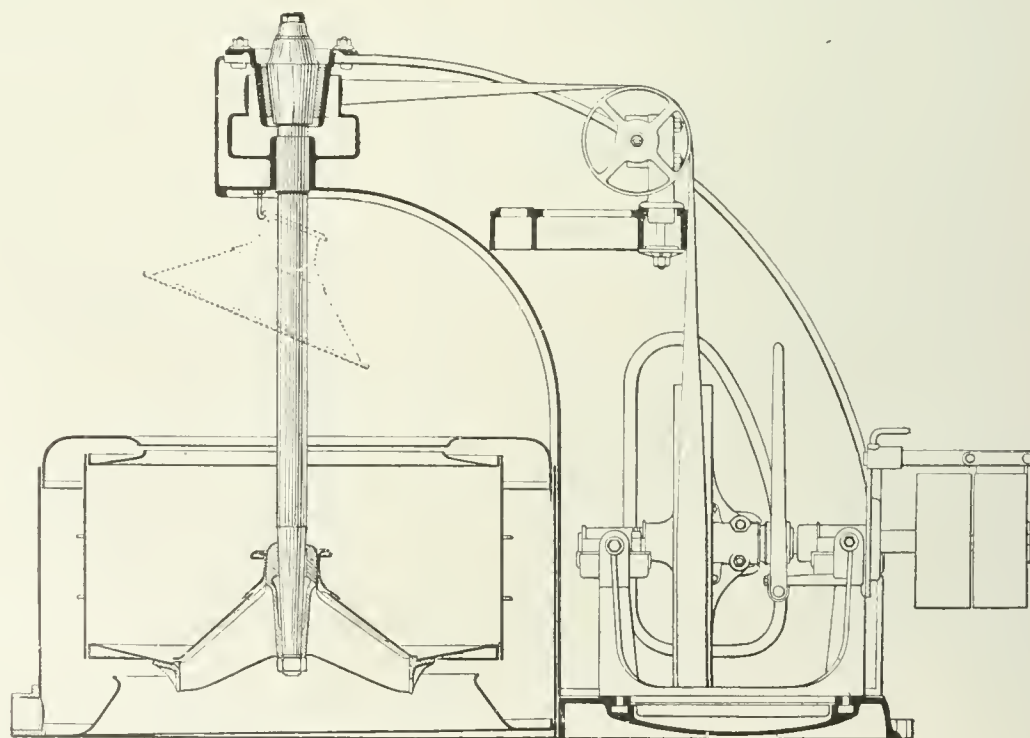
Fig. 4.

First we have the under-driven type. In this the bearing box can be supported either by a conical rubber buffer, as will be described later, or by plain rubber rings, of which one or two are placed on either side of a flange on the bearing box ; the whole is fitted into a kind of spigot in the base plate, and secured in any convenient manner. Here an arrangement must be provided for tightening up the rubber as required, but, as we shall see very shortly, care must be taken not to overdo this.

Another type of suspension which is familiar in the under-driven machines consists of a number of rods radiating from a pivoted bracket which carries the spindle and rests on a hemispherical bearing. The rods pass through the support for the outer pan and terminate in constraints, consisting of steel springs inside stiff rubber buffers.

The top-driven or Weston type is often fitted with a conoidal buffer, Fig. 4, though in some forms a straight-sided rubber cylinder serves very well. The former type is self-adjusting, and provides an almost perfect control, combined with great durability. Another satisfactory type has two rubber rings instead of a conoidal buffer. These have no adjustment, and are kept tight by the suspended weight.

The straight-sided buffer usually needs means of adjustment, and in one variety it is surrounded by a steel ring, which is conoidal on its external face. It is cut through, or partially cut through, at several points, so that its diameter can be varied by drawing it down by means of bolts on to a conoidal outer support. This allows a nice adjustment of the buffer pull, when exceptional loads or baskets are being dealt with. It is, however, subject, as all adjustable buffers must be, to the vagaries of the operator (seldom instructed in the somewhat perverse mechanics of apparatus of this kind), who usually falls into the heresy of thinking that if he has trouble in getting sweet acceleration, it will be of necessity improved by tightening up the buffer.

(b) Principles of Self-Balancing Machines

Let us now consider how it is possible for a machine to balance itself. We may begin by examining some properties of elastically supported bodies which are free to vibrate. Take, for instance, a four-pound kitchen weight hanging from a three-foot piece of doubled catapult elastic, the ends of which are held in the hand. If the weight is simply displaced and allowed to vibrate by itself, it does this at a certain rate, however big the vibration. This is the critical speed. If the hand is moved up and down slower than this the weight follows, and moves up and down as the hand moves up and down. Moreover, as the speed is gradually increased, the length of the up and down movement becomes greater and greater ; and it is impossible to speed up to just the same rate as the weight itself vibrates if left free, for the motion would then be so violent as to break the elastic.

A curious thing happens when the speed is increased still further. The weight no longer follows, but lags half-way behind, moving down as the hand moves up and vice versa. Its motion, moreover, becomes less and less, and if the hand is jerked up and down very rapidly the weight scarcely moves at all. Even if the hand moves a long way up and down—so long as it does so sufficiently quickly—the elastic simply stretches and the weight stays very nearly still. This exemplifies the natural motion of any elastic system when caused to vibrate more rapidly than its natural speed. There is another curious point, for if some friction is imposed on the weight by making it, for instance, vibrate in water, its movements become much less violent even at the critical speed.

Now let us turn to the " free spindle " centrifugal. The basket corresponds to the weight, the buffer to the elastic, the out of balance weight to the pull of the hand, and the speed of the machine to the rate at which the hand is moved. Such a centrifugal has a critical speed at which it vibrates, just as the weight did, and this speed has a close relation to the rate at which the basket would swing across and back again if it were pulled hard to one side and let go. At low speed the spindle is straight, or nearly so, and the out-of-balance weight is pulling it outwards. The deflection increases till the critical speed is reached. At a little above the critical speed, the spindle is still a long way out of truth, but the basket lags behind in the manner common to all forced vibrations, and the out-of-balance weight is now half a turn in front. The spindle tends to move back as the speed gets higher, and at high speeds it returns so far that its centre of gravity comes practically into the vertical line in which the spindle normally would be. Unless the basket is very badly loaded the centre of gravity will only be perhaps $\frac{1}{8}$ in. or less from the centre line of the spindle, so it will be seen that very little side strain is placed on the bearings.

Fig. 5 indicates the manner in which the spindle deflection varies at different speeds. The latter are shown at the bottom of the diagram, and the amount of deflection at the side. There are four curves showing the result of tightening up the buffer. It is assumed that the basket weighs 1247 lb. and is one-tenth of an inch out of truth. The buffer pull is taken at 25 lb., 100 lb., 400 lb. and 1600 lb. per inch deflection at the centre of gravity. The deflections are calculated by equating the centrifugal force, at the centre of gravity of the basket, to the buffer pull, on the assumption that the machine is given time to attain its full deflection, and that the whole mass of the basket may be considered as acting at its centre of gravity. Observe how increasingly out of truth the basket tends to run near the critical speed—if kept running at the latter its displacement would be indefinitely great and, if it is not actually so, it is because there is sufficient acceleration so that it has no time to attain anything like the full value at this point. The presence of friction also limits the deflection at the critical speed, though at some cost in smooth running at high speed if too much be present. It also affects the position of the unbalanced load, which is now between 90° and 180° in advance above the critical speed, and 0° to 90° below it.

Increasing the spindle length decreases the buffer

pull on the basket, and the critical speed increases as the square root of the restoring force. The range of speed during which any given deflection (say twice the eccentricity) tends to be exceeded varies in proportion to the critical speed which is therefore a very useful criterion.

Providing that the moment of inertia of the basket about the spindle equals that about a diameter through the centre of gravity, it will behave as though its whole mass were concentrated at the centre of gravity. Excess about the spindle acts in aid of the buffer, and increases the critical or bumping period. Excess about the diameter has the reverse effect. In most cases, especially if the spindle is not too short, the difference made is of little consequence.

There is a very interesting way of making an unevenly loaded machine run in almost perfect balance, practically as soon as it passes the critical

method consists in arranging a number of rings in a casing at the centre of the basket, allowing them sufficient freedom to arrange themselves in opposition. Unfortunately such devices are not wholly an un-mixed blessing, as the movable weight is apt to work with the unbalanced weight till the critical point is passed, but it certainly makes for sweeter running at high speeds. For similar reasons it is difficult or impossible for any " free spindle " machine to run steadily, below the critical speed, if it contains any great quantity of loose liquor. Under these conditions the unbalanced weight is outside, and the liquor flies to the same side, and continually adds to it. Filling in such cases must not be attempted till sufficient speed has been attained.

In addition to the " whirling " or vibrating motion, there is also a motion of precession, in which the spindle—in addition to rotating—also moves (usually relatively slowly) in a circle. This may
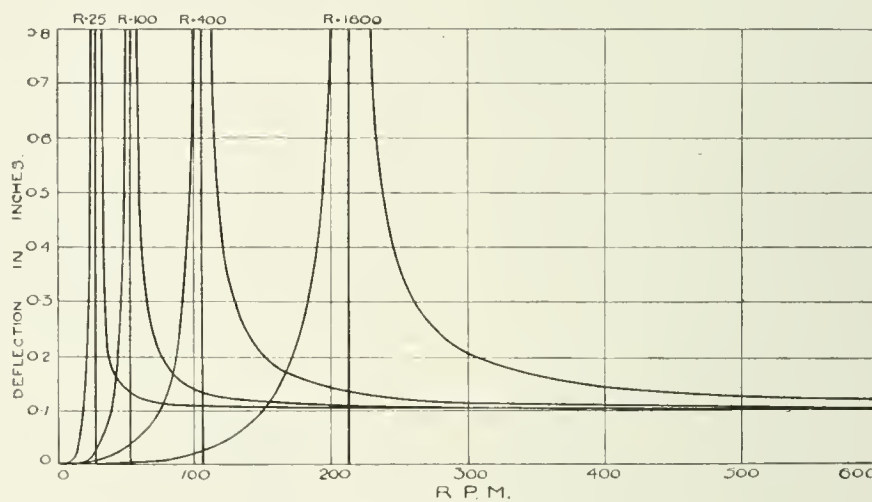
Deflection at Mass Centre of a 40 in. Basket.

Fig. 5.

speed. It will be observed that above this speed, the part of the basket that is farthest away from the vertical axis of spin is the portion opposite to the out-of-balance weight. It follows that if there were anything in the basket that was free to move, it would try to get as far away from the centre as possible and would place itself at this part. If, for instance, one puts some steel balls in an empty basket and fastens a weight on one side to make it out of truth, there comes a moment when the balls fly apart with a click and arrange themselves just to counterbalance the uneven weight. If this experiment is tried, a little grease must be put in with the balls, or they will not steady down very readily. In some types of machines compensation is secured by fastening circular tubes containing mercury to the basket. The mercury, like the balls, tends to fly away to the lightest side of the basket and helps to bring it into balance. Steadying friction is here provided by the comparatively small diameter of the tubes in which the mercury is enclosed. Another

occur at any speed, but most definately at the faster ones.

It may be seen even if loading be apparently quite even, and in some cases it will grow until the machine is stopped, or until the spindle is controlled by the hand or a bar being laid against it.

How does this motion arise ?

The swirl of the air may overcome the resistance due to buffer friction, thus promoting an increasing precession. For this reason baffles are sometimes fitted inside the casing, to help to break up the air currents. Another cause may be undue friction in the flexible connection or clutch transmitting the drive to the spindle.

In general, whirling vibrations are apt to appear more easily in short spindled machines, especially if the buffers have been adjusted too stiffly. Pre-cession, on the contrary, is seen more often in long spindles, particularly if the buffers are worn or out of adjustment.

Speaking generally, therefore, a good buffer must

not have too strong a pull, but enough to centre the spindle properly and not very much more. It must also have a sufficiency of friction or drag to hinder any precession, so that springs (which have little friction) are not suitable for buffers unless special precautions are taken to provide some friction. Rubber, however, has plenty of internal friction, and a properly designed rubber buffer will ordinarily do all that is necessary. It will be realised, of course, that if the drag is made unnecessarily great, some of the advantage of having a self-balancing machine may be lost, due to the additional wear and tear. The proportions of the machine, of course, help, but there is a simple test which is an almost infallible guide. One should be able to pull the basket a little to one side without great exertion and without feeling the buffer to be uncertain. When let go it should merely travel over to the far side, and back to the centre and stop practically dead. Other baskets may run satisfactorily when loaded by a skilled hand, but usually only the type here described can be run without trouble by the lowest class of labour.

Top driven machines are often fitted with a bar attached more or less firmly to the outer casing. This has usually a central notch to fit half round the spindle, and it can be rapidly swung into place to control precession if this is set up. In this case it is not the direct centering effort that helps, but the tangential drag imposed on the precession.

It was once thought that a self-balancing machine could not run if its basket had a greater depth than diameter. Rotating bodies, if left to themselves, of course spin about their axis of greatest inertia, but a centrifugal spindle does not accommodate itself to this action, which in effect merely decreases the buffer stiffness. Such baskets present difficulties in design, however, if uneven loading can occur, as two opposite inequalities, one at either end of the basket, would constitute a couple having a very long arm, and the basket and spindle would be subject to great stresses. This, however, is merely a problem of mechanics and cost.

Swirling of liquid loads is perhaps more liable to occur in baskets of which the depth ranges between one and three times the diameter, but is quite capable of control by suitable design. Baskets of every proportion have been built to take liquid and semi-liquid loads.

(c) INFLUENCE OF SUNDRY FACTORS ON THE MATERIAL UNDER TREATMENT.

We can now consider with advantage the behaviour of material when treated in a centrifugal dryer, and it is convenient to take the various factors in the following order :—

(1) Centrifugal effect, and position of sample in basket.
(2) Time.
(3) Size of grain.
(4) Capillarity and dilatancy.
(5) Viscosity of mother liquor.
(6) Temperature, and air drying.
(7) Other factors.

It may safely be said that the influence of centrifugal effect, or time, falls off rapidly, in the case of most materials, once a certain minimum has been passed.

Drakeley and Williams (J.S.C.I., November 15, 1922) showed that wide variations in speeds might make very little difference to the " efficiency " of the machine, in the case of sodium sulphite crystals and other products. In general the writer's investigations closely confirm their conclusions, but in their investigations they took the " efficiency " as the proportion which the remaining liquor per 100 lb. of solids bore to the original quantity as it entered into the machine. It is, however, the last traces that count, and the real criterion from the point of view of the after treatment of the material is the wetness of the whizzed product, without reference to the original amount of liquor. This may be expressed as a percentage, but a more instructive figure is the pounds of free surface moisture left per 100 lb. of dry product.

In testing one product, having the weight and consistency of a loamy sand, the remaining moisture was 13·2 per cent. (15·2 on 100 dry), when the effect was 626 units and 15·8 per cent. (18·8 on 100 dry) where the effect was reduced to 218 units. One test on a cotton fabric gave 33 per cent. (49·3 on 100 dry) for an effect of 640 units, 38 per cent. (61·3 on 100 dry) for 412 units and 41 per cent. (69·5 on 100 dry) for 180 units.

The results found are in rough agreement with the rule

$$M \log E = K$$

where M is the remaining liquor, in units per 100 of dry product, E the centrifugal effect, and K a constant.

In many cases it is well to follow the usual filter press practice and fill at low pressures or speeds, and bring them up only when the bulk of the liquor has been removed. This is effective in bringing away the last traces of moisture, and has the advantage that great quantities do not have to flow through a closely packed mass. This consideration comes specially into account when products in suspension in a thin slurry are of distinctly higher specific gravity than the surrounding liquor. If it is attempted under such conditions to bring the machine rapidly to speed, the solids would immediately be thrown to the side where they compact and often form a very effective barrier to the passage of the liquor. Machines are therefore often run at low speeds during the filling operation, and this, moreover, has the advantage of minimising splash. Variable speeds for driving the machine are not usually necessary for this purpose, and in belt or electrically driven machines, where a friction clutch is used, the power absorbed by the incoming liquor will often keep the speed down for the requisite period, or the drive may be adjusted by hand as required. When this is done, however, care must be taken to avoid filling at speeds too near the critical ones in the case of a self-balancing machine, or trouble will occur.

The question of distribution of the moisture in the various portions of the load is of interest. The wettest part is generally next to the basket wall, and the driest on the inner wall of the load. In

handling fabrics, however, some very curious figures come to hand, and in such cases the driest portion is usually intermediate in position. For instance, in a ten minutes run in a 36 in. hydro dealing with cotton sheets at an extractive power of about 440 the moistures were as follows :—

MOISTURE PERCENTAGES

Position	Ten minutes run %	Twenty minutes run %
Near rim	43	40
Intermediate ..	32	31
At interior	36	35

Such results were found fairly regularly, but not invariably, when handling textiles.

A somewhat similar effect was obtained in a run on caustic mud, which had been settled and was scooped into the machine containing 46 per cent. of moisture. After spinning for 8 minutes in a 48 in.

were remarkably even and regular, but increased slightly from inside outwards and from top to bottom. The average size of the crystals at each point approximated closely to 0·2 mm., the largest being 0·5 mm.

The following further tests were made to ascertain the influence of the length of the operation on the dryness. Here again some curious inversions appear. The salt was run in as a hot slurry.

MOISTURE PERCENTAGES

Time after reaching full speed Mins.	Position (See Fig. 6) 4 %	5 %	6 %	Average moisture %
0 ..	2·56 ..	7·18 ..	5·05 ..	5·14
3½ ..	2·00 ..	3·76 ..	5·39 ..	3·72
7 ..	1·44 ..	2·84 ..	2·48 ..	2·25
14 ..	1·31 ..	2·05 ..	2·75 ..	2·03

Size of grain is always a very important factor. Ordinary caustic mud, in which there are many

1. 2·22%	2. 2·35%	3. 2·49%
4. 2·29%	5. 2·40%	6. 2·84%
7. 2·37%	8. 2·58%	9. 4·67%
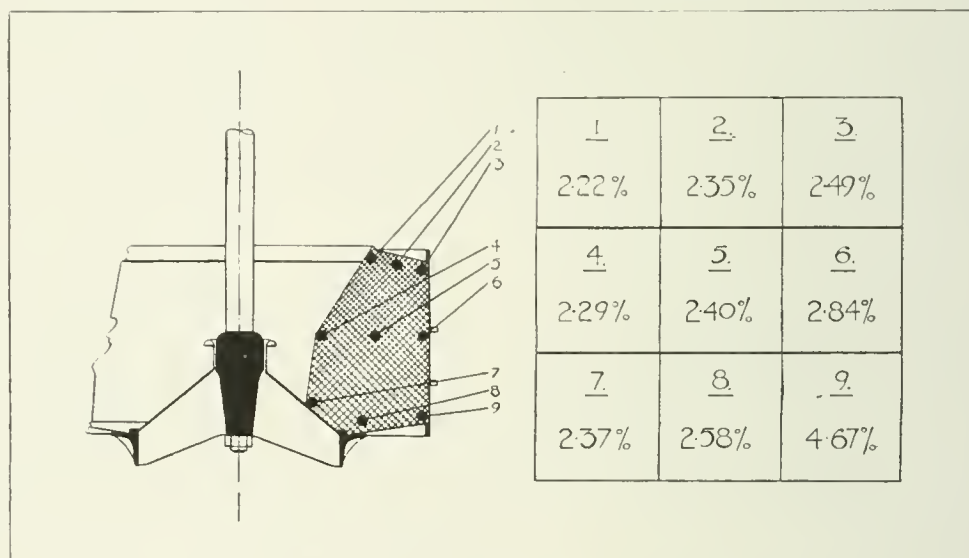
Diagram illustrating how moisture contents vary with position in basket.

FIG. 6.

machine which accelerated slowly to an effect of 600, the dried mud formed a layer of 4½ in. deep, and samples taken from a position half-way down the basket gave 33 per cent. moisture for the inner surface layer, 37 per cent. for the intermediate one, and 33·3 per cent. for that next the rim. These results, however, were peculiar to these conditions, and for a run of double the length the corresponding figures were 23, 26·1 and 33·2 per cent. In an 8 minutes' run, with a layer of half the thickness, the figures became 21·5, 28·3 and 35 per cent. This is interesting as showing the effect of time and thickness, and the care needed in sampling if a true indication of the average moisture is desired, in the case of such muds.

Some interesting tests were made by the writer on salt, spun in a 48 in. machine, with a view to determining what was an average sample. The results are indicated in Fig. 6, and it will be observed that the moistures, after a run of 7 minutes at full speed,

particles of 2μ (0·002 mm.) and downwards, will contain, under normal conditions, 25 to 35 per cent. of liquor, fine Epsom salts, vacuum salt or sulphate of ammonia (say 0·2 mm.—0·5 mm.) come down to about 3 to 4 per cent., large sulphate of ammonia crystals or the grains of crystal sugar (say 1 mm.—3 mm.) come down to about 1 to 1½ per cent., while extremely large grains, say over 5 mm. in size, like certain crystals of copper sulphate, may retain only a fraction of 1 per cent. of moisture. If the liquid flows readily 7 to 15 minutes generally suffice for the finer sludges, 4 to 7 minutes for medium crystals, and 2 to 4 minutes for very open ones. Good crystallisation, therefore, is the first step toward efficient results in the centrifugal. One firm producing naphthaline were able to improve its melting point by 2° C. by attention to this point.

Drakely and Martin (*J.S.C.I.*, December 31, 1921) consider, among other things, the effect of the pressure and so forth on the *manner of the piling*

of the grains, but their results were negative. One would not expect the effect of *dilatancy* to be exhibited in any remarkable manner, but fine grains of any hard substance do not usually form their most compact arrangement under centrifugal force.

Clearly *capillarity* plays its part. If the capilliary passages are sufficiently small, it will be quite impossible to remove the interstitial liquor by any ordinary centrifugal force. If the particles are very small this will apply to the whole thickness of wall, otherwise to a layer at the rim of a depth determined by the size of the passages and the force employed. So long, however, as the relative surface tensions permit the liquor to wet the solids, there will always tend to be a thin film over the surface of the particles, even if the interspaces are empty. The thickness of this film will depend on the viscosity, and it will be continuous throughout the mass, extending unbroken from the rim to the innermost layer and from one particle to another.

The effects of dilatancy and capillarity may clearly serve as a partial explanation of the curious results for moisture percentages in different layers in the basket, as noticed above.

Viscosity is probably next in importance to size of grain, for if it is high, not only will a long time be required to pass any quantity of liquor through the basket, but also the retained liquor will be very seriously increased. It is, therefore, well to reduce viscosity by *heating* or any other practical means available to an extent dependent on the temperature viscosity curve of the liquor in question.

It may be worth considering how far air-drying comes into question, and here again the effect is not as great as one would suppose, except when large crystals of open structure are being treated, especially if these are fed into the machine as a hot slurry. In such cases, however, samples taken from the machine will be slightly damper than those collected from the belt after the material has been discharged. In the case of sulphate of ammonia fed as a hot slurry, carefully averaged samples from the basket at one works showed 2·12 per cent., while after the material had been dropped out of the machine, elevated to a conveying belt and carried to the drier inlet, the average sample was approximately 1·29 per cent.

Washing is an operation often performed in the centrifugal. All kinds of methods are in use, such as tipping in with a bucket ; pouring with a watering-can and rose, spraying from one or more nozzles, etc. For good results it is essential to spray the wash liquor on as finely and gently as possible. Otherwise it will cut channels in the wall of material, and if too much reaches any point it will flow upwards over the surface, instead of percolating evenly through the cake. Good washing is obtainable, though in many cases it is not so good as is generally supposed, on account of the difficulty in obtaining an equal spraying of all parts of the material, combined with the varying slope, permeability and thickness of the wall. It is a good thing if a measured quantity can be used for each wash, and automatic devices are obtainable which secure this. One user employed about 1 to 1½ gallons of water to wash a load containing 3½ to 4 cwts. of granulated sugar. Another used 1 gallon of hot water to wash about 6 cwts. of sulphate of ammonia, followed by about 8 gallons of an alkaline liquid for purposes of neutralisation. Whizzed anthracene and naphthaline were found by one manufacturer to improve somewhat if washed with a little hot water.

(d) CAPACITIES.

We may now consider the extent to which the average basket can be loaded. Makers often give two capacities "net" and "gross." The "net" capacity is the volume under the basket rim and regulates the maximum amount of liquid load that could be in the basket at any moment when the latter is running. Gravity then is only an insignificant portion of the total force and the liquid load stands in a wall which differs little from the vertical. The "gross" capacity represents the total space available for packing in material. For instance, it is usual to fill a laundry centrifugal right up to its gross capacity, but as soon as the machine has been running a minute or two the whole of the load is compressed under the rim.

(e) LABOUR AND OUTPUT

We now come to the question of labour and output. Here a good deal depends upon the class of man employed, and the nature of the supervision. Still more depends on the conveniences provided for filling the machine, and for disposing of the spun product.

Although no special methods are possible when dealing with fabrics and textiles generally, yet loading and unloading are often accomplished fairly quickly when labour of not too low a class is to hand. Ten 10-lb. hanks of cotton yarn (weighing, say, 30 lb. each when wet) would be loaded, or unloaded, from a 48-in. basket in 2 minutes or under. A 36-in. machine (7 cubic feet gross) would take about 4 to 6 minutes to load and pack thoroughly with articles of laundry, and 3 minutes to unload.

Soluble nitro-cotton in one instance was run in by means of a circulating pipe, having branches arranged to swivel over the machines. These each ended in four branched outlets, over which were placed the mouths of bags into which the pulp was run. The mouths were tied when the bags were full, and the latter packed into the basket while the filling pipes were swung back.

Filling a 48-in. machine (7 cubic feet nett 16 cubic feet gross capacity) with materials spaded over the side—partly drained caustic sludge, anthracene, naphthaline, etc., will take about 4 to 7 minutes and discharging 10 to 20 minutes, according to the solidity of the spun material. Bottom discharge doors appreciably reduce this time in the case of easily broken down products, such as Epsom Salts, for which in a 36-in. machine 14 in. deep (3½ to 4 cwts.) only about 5 to 6 minutes would be needed.

When the product can be run into the machine much time is saved. For thick slurries which spread evenly 1 minute to 2 minutes is usually ample for almost any reasonable size. If the load has to be evened up, another minute or so will be taken; even

for weak slurries 5 to 10 minutes is ample in almost every case.

Self-discharging machines working on suitable products need only half-a-minute to a minute to empty. Ordinary forms of central bottom discharge vary from about $1\frac{1}{2}$ to 2 minutes for products which drop away easily in, say, a 48-in. machine 24 in. deep, to 7 or 8 minutes for such stuff as caustic mud.

It must not be overlooked that the rate of output found under observation, for a single run, or for an hour, may not always be maintained. It may be closely approximated if the work of filling and discharging is light, and one man looks after one, or at most two, machines, and some judgment must be used in making any estimate.

It now may be interesting to take a few concrete cases.

In a battery of 48 in. self discharging machines working on raw sugar, two timed operations were as follows :—

Test		Min.		Min.
Charging	..	$\frac{1}{2}$	..	$\frac{1}{4}$
Wash or dry	..	4	..	3
Brake	..	$\frac{1}{2}$	..	$\frac{1}{2}$
Empty	..	1	..	$\frac{1}{2}$
Total	..	6	..	$4\frac{1}{4}$

12-15 operations per hour are usual under such conditions, where great dryness is not essential, but of course such machines are suitable for relatively few products, as has been explained.

On a battery of 6-42-in. centre discharge machines handling granulated sugar five men were required—one charging, one washing and blueing, and three digging down. Times were as follows :—

				Min.
Fill and wash	..	..	..	2
Drying	..	..	..	3
Discharge and restart	..	..	$6\frac{1}{2}$

This gave a cycle of $11\frac{1}{2}$ minutes, producing some $3\frac{1}{2}$ to 4 cwts. of spun sugar. The total output corresponded very fairly to this.

In a plant handling caustic mud, consisting of six machines 48 in. dia. x 24 in. deep with centre discharge, the following figures would hold for individual runs :—

				Min.
Filling	..	..	..	7–8
Drying	..	..	..	15
Emptying	..	..	..	6–7

Thus the average length of a unit operation would be, say, 30 minutes.

It was economical in this case to have two men to work this plant, and it would appear, since the machines did not need the whole of their attention while filling, that in theory this average rate should determine the output, which would mean ninety-six operations per eight-hour shift. The average, however, was sixty operations, being roughly three tons of spun material per hour.

In another plant handling sulphate of ammonia there were a number of units, each having a single centrifugal to take the salt as it came from the saturator. The baskets were 42 in. diameter, but only 17 in. deep, and the average load was about 3 cwt., with a maximum of about 4 cwt. of whizzed salt. One man attended to the saturator and centrifugal, the dried product from which fell on to the boot of an elevator, whence it passed to conveyors and rotary driers. Here a timed operation was as follows :—

				Min.
Filling	..	..	..	3
Drying	..	..	..	3
Stop, empty, restart	..	..	4
Total, about	..	..	..	10

Here the average for any machine kept up fairly well to the unit operation, so long as sufficient salt was available, but was apt to drop a little through various causes when the whole battery was considered over a shift.

In a similar plant having 42-in. baskets $23\frac{1}{2}$-in. deep, in which the product was washed and neutralised, only about $2\frac{1}{2}$-3 operations per hour were worked, or say 20 per shift. Each operation gave some 5 or 6 cwts. of whizzed salt, and here also the ratio of output was maintained.

Three 40-in. self-balancing machines with lifting out baskets, working 57 hours per week dealt with 2887 tons of swarf and oil, extracting 875 tons of oil per year. Two to three operations would be worked per machine per hour, and two men would be necessary. In this case a load of short chips was 5 cwt., and gave 20 gallons of oil, but a load of long chips was only 2 cwt., yielding $2\frac{1}{4}$ gallons of oil.

Two machines were added later and worked on fine chips, the baskets being raised by hand. The times for charging are given in column A below. The first three machines were then confined to coarse chips ; the baskets were raised and moved on overhead rails to the discharge point by electricity. The times are given below in column B.

		Plant			
		A		B	
		Hand lifting fine chips		Motor lifting coarse chips	
		Min.	Sec.	Min.	Sec.
Fill 40 in. basket	..	2	26	3	38
Place in machine	..	1	28	0	47
Whiz	..	10	0	14	0
Remove basket	..	1	35	1	0
Empty and return	..	1	10	1	40
Total	..	16	39	21	5
Time per cycle if spare basket used	..	13	3	15	47

The time per cycle is less than the total, because a spare basket can be put in already charged as soon as the other comes out. The coarser chips are more awkward to handle than the fine chips, and give much less oil even though spun for some minutes longer.

Before we leave the question of capacities, output, etc., the following table may be of interest. The figures are obtained from ordinary commercial practice, but it will be realised that they are given as a guide and not as a standard. They must necessarily be influenced by the quality of the product,

the efficiency of the crystallisation and so forth, according to the particular condition in each individual problem.

Material	Approx. spinning moisture content, etc., after spinning	Approximate spinning time from start to application of brake Mins.	Approx. density of product lb. per cub. ft.	How reckoned
Anthracene ..	40–45% anthracine content	*30	60	After spinning.
Cotton sheets ..	30–36%	7–10	12–15	Before spinning. Dry fibre as loaded.
Cotton yarn ..	32%	5–8	†16	Dry fibre per cu. ft. net.
Cotton (soluble nitro) ..	31%	6–15	†10	Do. do.
Caustic mud ..	25–35%	15	90	After spinning.
Epsom salts .. (Medium crystals)	3–4%	2	60	,, ,,
Naphthaline ..74°–77° C. (m.p.)		*30	56	,, ,,
Sodium benzene sulphonate ..	30%	5	50	,, ,,
Sugar (granulated) ..	2–3%	*3	56	,, ,,
Sugar (crystal)..	1½%	*2–3	56	,, ,,
Sulphate of ammonia (large crystals) ..	1–1½%	*3 7	52	,, ,,
Ditto (small crystals)	2½–3½%	*7–10	52	,, ,,
Vacuum salt ..	3–5%	5–7	58	,, ,,
Wool (blankets).	40–45%	3–5	7	As laid in centrifugal (dry fibre only).

NOTE.—The spinning periods do not include time of filling.

* These periods include an allowance for washing.

† These figures represent loading factors rather than densities.

Various types of automatic centrifugals are being introduced. At present little data is available, and such machines require special care in construction and are apt to be costly.

Various types of continuous centrifugals have been developed. One has worms to elevate the spun product out of the basket. Others have filtering compartments, fed from a distributor with doors which are opened for discharging, at regular intervals, by cams or by oil pressure. Others have a separating lip, under which the spun solids, of a plastic nature, can flow, and discharge continuously, forming their own seal against the liquid portions which accumulate above them and flow over another lip.

II. CENTRIFUGAL SEPARATORS

(a) DISCUSSION OF FEATURES PRESENTED BY EMULSIONS AND SUSPENSIONS

We now come to another class of centrifugals, known as subsiders or separators, which have no holes in their baskets. They can be used to remove from liquids solid particles so fine that they would choke any filter cloth, or for separating two liquids one from another, such as water from tar. If left to separate by gravity in a tank, such materials might require many hours or even days, but in separators a few minutes or even seconds will often suffice. It will be interesting to examine some of the properties of suspensions and emulsions, such as are dealt with in these machines.

Almost any substance, liquid or solid, will form a permanent emulsion or suspension in another liquid if it is broken up sufficiently finely and other conditions are right. One hundredth of a centimetre is a large size for suspended particles, and these may be a thousand, or even ten thousand times smaller still, before the highest practicable force will cease to effect a reasonable separation

If we take 1 centimetre cube of material and cut it up into cubes of, say, one-thousandth of a centimetre in size, we increase the surface in proportion to weight by one thousand times, and so it will be seen, in the case of very small particles, the amount of surface in contact with the liquid may be very great indeed. Now the surface of a liquid differs from the rest and behaves in some respects much like a stretched elastic skin, and is in fact the playground of very powerful forces. When we have large numbers of very small particles the liquid surface has to stretch all round them. This involves a large increase in the surface energy, and forces are brought into being which tend to bring the particles together reducing the surface, and consequently, the energy taken up by it.

Such substances as soap, gums, gelatine, albumen, tannic acid and many others are apt to reduce the surface energy of liquids, and consequently decrease the tendency for the suspended particles or drops to agglomerate. It is thought that they tend also to form a kind of protective film on the surface of the particles, though the actual process can hardly be indicated in a few words.

Low surface tension not only increases wetting power, but tends to separate adhering particles and films. Everyone is familiar with the action of soap in assisting the dirt on clothes to break up and form an emulsion. An interesting experiment is to take a glass tube with a fine nozzle at the end, and use it to pass oil into water. Drops of oil will come out, and when they get big enough they will break away and float to the surface. If about one part in a hundred of soap is put into the water the drops break away much more quickly and are smaller, so that a given amount of oil is split up into many more droplets. The precise action of the various emulsifying agents is highly complicated ; but the main thing to be grasped is that very small traces of these substances may make it much more difficult for particles to agglomerate and settle, and such small traces are apt to be present where they could be most dispensed with.

It is often necessary to neutralise or destroy the emulsifying agent, or to employ other means for causing the suspended particles to coalesce, before the suspension or emulsion can be dealt with at all. The method used has to be adapted to the particular circumstances of the case, and would require much space to describe in any helpful detail. It is sufficient to say that digestion at high temperature is often useful, and tends to cause larger particles to grow at the expense of the small ones, providing solubility increases with temperature ; the addition

of small quantities of acid may help ; salting out, is often effective, while a more recondite method is to employ an agent tending to reverse the type of emulsion. Much study and research is often needed to find a satisfactory and economical method.

Even when action of this kind has been taken, much time may be needed to settle by gravity, as the particles may remain so small and light in proportion to the amount of surface they possess that they are held up by the liquid.

Where the particles are small (but not excessively so), the resistance to fall mainly arises from the viscosity of the liquid, which exerts an effect proportional to the velocity, and Stokes law is obeyed while the particles are so far apart that they do not interfere with each other. When they become more concentrated the resistance is probably of the capillary type, proportional to the velocity at any stage, but rising rapidly from stage to stage as
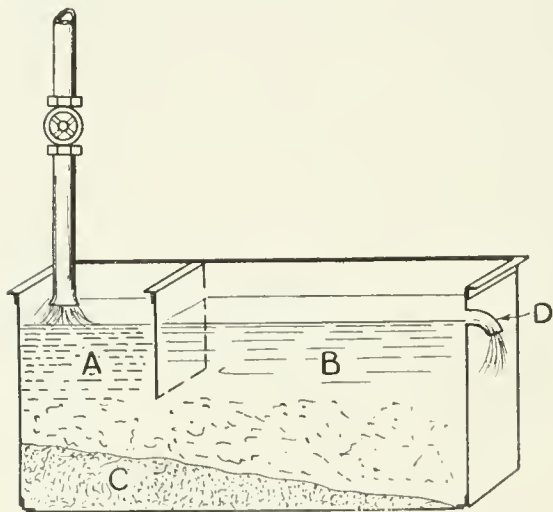

FIG. 7.

concentration increases, and the passages between the particles become narrower. There is, according to some authorities, a golden rule connecting these two phases, namely, that for a given suspension the time taken to sink from one given average concentration to another is proportional to the amount of suspended matter per unit settling area, or to the depth, for suspensions of the same critical strength (cf. " Principles of Chemical Engineering," Walker, Lewis and McAdams, p. 332).

(b) PRINCIPLES OF CENTRIFUGAL SEPARATION.

The action of a centrifugal separator may best be explained by considering a tank into which a mixture of water and solids is run at one end, where a baffle is fixed to distribute the flow. (Fig. 7.) The coarser or heavier solids go to the bottom at once, and the somewhat lighter ones more gradually, so that if the tank is long enough the water is clear as it flows out at the other end, and all the solids which can sink have settled to the bottom. In a centrifugal separator we have a basket with no perforations.

The liquid is fed to the bottom through a

baffle or distributing ring, which brings the water up to the basket speed as it enters. Instead of gravity we have centrifugal force which makes the liquid form a substantially vertical wall level with the lip of the basket. The particles of dirt sink to the side while clear liquor goes over the top. If we run a mixture of two liquids into a tank with a baffle plate in the middle, the heavy liquid will sink, and pass under the baffle, where it rises again owing to being pushed up by the head of liquid on the other side. If the tank is correctly proportioned none of the light liquid will pass below the baffle, but it will stay above the heavier one, so that spouts can be arranged one on either side, which will deliver almost pure heavy and almost pure light liquid respectively. The purity depends on the ease with which the two components separate, and one portion may retain traces of the other depending upon the factors of time, force, viscosity, temperature, the presence of protecting agents, etc. An interesting point is that the heavy liquid will balance a rather deeper column of light liquid, so that the latter could be run off at a higher level.

Here again the action may be stimulated and intensified in a centrifugal separator. The mixture is fed in, often under a distributing plate, and rises towards the top lip, where there is a circular ring or plate corresponding to the separating baffle in the tank. The diameters of the rim of the basket and the separating ring are adjusted to the specific gravities of the mixture being dealt with, and its components. The heavier liquid passes under the ring and away over the outer lip, the lighter one may be taken away by an adjustable skimming pipe, or the separating ring may be extended upwards to form a lip from which it flows to a separate catch pan.

If the diameters of lip and separating ring are very nearly equal, heavy liquor cannot overflow until a considerable amount has collected on the light liquor side so that sufficient head is available to counterbalance that due to pure heavy liquor. This will be understood if we consider a U tube with water in one arm, and mercury in the other. Unless there is a column of water much higher than that of the mercury the latter will flow into the water side until it has made good the defect in head due to the short water column.

Instead of heavy liquor there may be a deep column of partially separated liquor, which may pass to the light liquor overflow.

If on the other hand the difference in diameter of the plate and lip is the maximum permitted by the relative specific gravities, light liquor must collect till the light liquor side is entirely filled, before any overflow can take place. Unseparated liquor cannot form a deep enough column to overflow on account of its greater specific gravity, although it may pass to the heavy liquor discharge if any overfeeding or disturbance takes place. The designer can therefore adjust the machine by a simple alteration of the diameter of plate or overflow. These diameters must be such that the liquid mass per unit height shall be equal above and below the separating plate. The layer between plate and wall does not count.

The capacity of settling and separating tanks

depends primarily on their surface area and not on their depth. If the tank is twice as deep, particles take twice as long to reach bottom, so that velocity is reduced in exact proportion. A deep tank of course can run longer before it must be cleared out, and since the velocity is smaller there may be less tendency to sweep settled portions away again.

The same principle applies to settling in a centrifugal separator, but may be disguised or hidden by various disturbing factors.

Where the viscous resistance of the fluid medium is the main factor delaying settlement, complete settling may be obtained in time by gravity. Centrifugal separators should then give a comparative speed of settling proportional to the centrifugal force exerted. Usually their efficiency is impaired, as against quiet gravity settling, by the effects of undue vibration, short circuiting, cross currents, etc., and the actual settling efficiency may be much less than the theoretical one. Clearly a reasonable freedom from vibration is of importance, if separators are to give their best results, though vibration may actually be of assistance in aiding particles to disentangle themselves providing its amplitude is small, and doubtless something will depend upon its frequency.

When the particles are very small, they become subject to the Brownian movement. Perrin has shown that this is analogous to molecular agitation, and that suspended particles exert an osmotic pressure, increasing as their size diminishes. In such cases complete settlement is often not possible by gravity, however long applied. The effect of high centrifugal force is then unique, as it overcomes such pressure to a very large extent, and unless the particles approach almost molecular fineness, will give an increase of concentration not obtainable by any other mechanical means.

An interesting mathematical study of this point has been made by Eugene Ayres (Trans. A.I.C.E., 1916, Vol. IX., 203).

(c) PRACTICAL SEPARATORS, WITH SUNDRY DATA.

The various forms of separators in ordinary commercial use may now be considered.

The plain under-driven type, without suspension gear, and fitted with a solid wall basket, is very useful for many purposes, and is notably used in the manufacture of starch. In one factory the starch, after a preliminary settling and washing, was mixed with water again and run into the centrifugal as an even mass. The settled product contained roughly 50 per cent. of water, the starch contents of a 48 in. basket 16 in. deep being approximately 1 cwt. or a little over. The maximum centrifugal effect was about 600, or, say, 470, at the inner liquor face and the settling occurred in a 5½ in. wall in 8—10 minutes. The total cycle took about 13 minutes, of which, say, 3 minutes were taken by unloading. The settled starch was sufficiently firm to be lifted out bodily. The supernatant liquor was not quite clear, and returned to the process. It may be removed by a skimming-pipe, and a second smaller batch of liquor treated before the deposit is taken out.

In some cases the subsider replaces the settling tanks, and in that case the pure starch and the impurer portions settle at different rates, which causes them to form into more or less distinct layers, which can be separated after removal by washing or cutting off the impure inner layer.

Another use is in the pre-treatment of wool-scourings to remove the coarser dirt before the liquor is passed to the small high-speed centrifugals for the separation of the grease.

Here the procedure followed is to run the liquor on to the bottom of the basket and allow the overflow to escape over the rim. The capacity depends upon the type of wool liquor being treated.

Machines with horizontal spindles are sometimes used.

Another type of centrifugal separator has an elongated basket, the usual size being 3 ft. diameter by approximately 4 ft. 6 in. long. This, of course, is suitable for higher capacities, and with it advantage can be taken of the fact that the coarser portions settle more quickly than the finer sludge and are separated from each other into fairly distinct layers, as the liquor flows downwards through the basket. The latter has an inner cage which carries removable plates. These form a kind of false wall all round the basket proper, and receive the deposited matter. At the conclusion of the operation, this cage is drawn up mechanically out of the basket, which is usually arranged below the floor level, and the plates taken out and replaced by clean ones. Cleaning of the plates occurs while the next operation is proceeding, and the strips of material can be cut across and packed according to their grades.

One man per machine will generally suffice both for filling and for cleaning the plates, but in the case of single units working on a short cycle, a boy may be needed as well.

Ten to eleven horse-power will run the machine, when 1000 to 1200 galls. per hour are running through. For accelerating it is usual to provide 20 h.-p. Clay slip would be fed in at the rate of approximately 1000 gallons per hour. The deposit would weigh about 680 lb., and its average moisture content would be 25 to 30 per cent. This might vary considerably between the coarse and fine portions.

Tests with this machine on starch, with a view to replacing the settling tanks, as described above, were conducted, and it was found that with a rate of feed of 1200 galls. per hour the starch left in the overflow was approximately 0·05 per cent. The amount of deposited solids at the end of an hour's run was about 600 lb. containing 45 per cent. moisture. The centrifugal effect was approximately 500 at the basket wall. The following times may be of interest :—

	Clay. Min.	Starch. Min.
Accelerating	2 ..	3½
Feed	15 ..	56
Stop: Lift Containers:	7 ..	14
Replace Plates: Restart		
Total	24 ..	73½

The machines hitherto described have had baskets of large diameter, and rarely exceeded 1000 to 1500 revs. per minute. We now come to speeds of from 6000 revs. to 40,000 revs. per minute, for basket sizes ranging from 12 in. diameter down to about 2 in. Perhaps the most familiar form is the ordinary milk-separator, which may be modified, as required, for various purposes.

Such a machine has a bowl of from 6 in. to 12 in. diameter, according to the size of the machine. This bowl is practically entirely filled with a number of metal cones or inverted dishes made out of thin metal plates, separated slightly from one another by distance pieces. These fit round the central portion of the bowl, and are held in their place by tongues which fit in corresponding notches. The liquor to be treated is fed down the inlet pipe, through the centres of the plates, rises in passages which are cut through the latter and passes to the spaces between them. The object of these plates is twofold. In the first place, their friction helps to bring the in-coming liquor up to speed very rapidly, and minimises the risk of any disturbance due to surging or undue vibration. Secondly, it acts as an extension of the separating surface, for any particle in the thin film which is contained between each pair has only a small distance to move to achieve its final separation. Thus, in a milk separator, the heavy skim milk will form a thin layer moving outwards on the under-face of each plate, and the lighter cream a similar layer moving inwards on the upper face of each plate. The milk comes into the outer space, passes beneath the separating lip and flows out of the lower outlet. The cream similarly passes on the inside of the separating plate and out of the upper lip, which is slightly smaller in diameter than that over which the milk overflows.

A certain amount of freedom has to be given to the spindles of these machines, because any eccentricity of the basket would otherwise become very marked at these high speeds. The drive is generally by means of a helical wheel which drives a worm on the spindle shaft. Machines of this kind take very little power and owe their separating efficiency largely to the very small distance which any particle has to traverse before separation takes place.

It is very usual to provide three discharge openings at the top of the machine, and three pans are arranged, one on top of the other to collect each type of discharge. The lower one will take the heavy liquid, the next one the light liquid, and the third serves to take any overflow if the machine is fed too fast or becomes choked. If the feed contains solids of heavier gravity, these collect on the basket wall in the dirt space, and from time to time the upper part must be unscrewed, the plates taken out, and the basket cleaned.

Sometimes it is possible to string the plates bodily on a piece of bent wire of special shape, rinse them in warm water, and return them to the centrifugal, and comparatively little time is lost. In many cases all that need be done is to remove the sediment on the wall, and allow that between the plates to be washed out by the fresh incoming liquid.

The following capacities relate to such a machine with a bowl 11 in. diameter, containing 42 to 43 plates, running at 6900 r.p.m. (effect—5400 approx. at basket wall).

	G.p.h.
Turbine lubricating oil	300
Diesel lubricating oil	150–200
Heavy Diesel fuel oil	250
Used transformer oil	150–200
New transformer oil	250–300

Smaller sizes have capacities down to about one-tenth of the above, varying roughly as the square of the total diameter, and directly as the number of plates.

In dealing with more difficult problems such as wool washing effluents, two machines are often used in series, one to scour the liquor of grease, and deliver a strong emulsion of grease and water, the second to deliver a water-free grease. In such cases various tests showed about 0·4 per cent. to 0·8 per cent. grease left in the effluent, the recoveries ranging from 25 per cent. up to 65 per cent. on crude liquors containing from 0·7 per cent. up to 1·2 per cent. of fat.

The power required for an 11-in. bowl is about 2 h.-p. for acceleration, and 1½ h.-p. for running. If two liquids are being handled running is continuous, but if sediment is present it must be interrupted now and then for cleaning. This will put the machine out of action for a total of, say, 12 minutes, though this depends somewhat on the facilities provided and on the skill of the operator.

There are many different adaptations of the general scheme. A similar bowl is used for removing dirt from milk prior to cream separation, but this contains no discs.

Some machines are made with filters of various types to remove the last traces of suspended matter in difficult clarifications.

Finally, we come to another class of machine, which is made in only two sizes—one 4 in. diameter by 36 in. long, and the other only 2 in. in diameter. The usual speed for the larger size is 17,000 revs. per minute, giving a centrifugal effect of about 16,950. It presents some very interesting points in construction, owing to the unusual shape of its basket. The latter is supported by a flexible spindle, carried on a ball bearing at its upper end. The lower end of the bowl has a spigot piece which is led through a controlling apparatus at the bottom. This is scarcely a bearing in the ordinary sense, but consists of an arrangement which closely surrounds the spigot, and can only move under considerable friction, without any elastic force being present. This frictional control is particularly necessary for the successful operation of a bowl having the dimensions and speed described, but gives very smooth running. The drive is transmitted to the upper end of the spindle by means of motor, belt or steam turbine.

The liquor is fed under a slight head through the spigot at the lower end of the bowl where fins bring it rapidly to speed. It then rises, in a vertical wall, to the top where there is a separating head adjusted to the specific gravities of the main liquid components. The heavier portion flows through passages on the out-

side of the separating head to the lower discharge opening, and the light portion on the inside to the upper discharge. There are some quite interesting modifications of usage. For instance, if the liquor contains particles of a lighter specific gravity, these will rise to the liquid surface much more quickly than they would under ordinary gravity conditions. A bowl with a separating plate having no centre opening is then used, so that the liquid passes away, while the light solid matter collects in a cylinder below the separating plate. At the conclusion of the run the bowl is lifted out, its bottom unscrewed and the solids removed. If there are also solids of heavier specific gravity present, these also will be thrown out on to the sides of the machine while the first action is going on. Problems of this kind occur during the clarification of some forms of glue. The suspended matters, in this case, are considered by most authorities to be truly colloidal and need to be made to coagulate by suitable reagents. As they agglomerate they are found to be of lighter specific gravity than the glue surrounding them and are caught at the centre of the machine in the manner described.

A slightly different problem is encountered when treating a heavy lubricating oil, known as heavy cylinder stock, for the removal of wax. This may now be passed through the centrifugal for wax removal, instead of employing the older system of cold settling. In this case, brine is fed into the machine with the oil, and forms a layer on the inside periphery of the basket. The wax, having a greater density than the oil, is immediately deposited on the surface of the brine, but being of a lighter gravity than the brine it floats on top of it, and is discharged with it, the brine acting as a carrier liquid. The oil, wax-free, is discharged from the third layer, and it has been found that the oil produced in this way will have a cold test as low as 19 to 20° F. as against 50° from older methods.

An interesting application is the treatment of heavy fuel oil for Diesel engines. While a " gas " of " diesel " oil has been used in the past, there has been a tendency lately to use a heavier oil known as " boiler " oil, having a specific gravity of 0·95

to 0·96. It was found to score the cylinder liners due to the high ash content, which was about 0·065. This could be brought down in a high-speed centrifugal to about 0·022—an amount similar to that of an ordinary Diesel oil. Such machines work quite well aboard ship, and pass some 250-350 gallons per hour, the bowl being changed over at the end of each watch. A separate overflow for any water is provided, so that in addition to freeing from ash security is given against water being injected into the engines.

Another interesting development is in handling a water-gas tar, in which oil forms a very intractable emulsion in water. A very inefficient separation is accomplished by gravity and heat and requires some nine to twelve months to complete. The finely divided carbon present acts as an emulsifying agent, and its density causes it to be removed almost immediately the emulsion enters the centrifugal. The latter is now free to separate with far greater ease at a rate of some 170 to 180 gallons per hour. The mucilage from linseed oil, which requires some 7–8 months for settlement, or 2 or 3 weeks if broken by a heat of 280–300° C., may be adsorbed by mixing in $\frac{1}{4}$ per cent. to $\frac{1}{2}$ per cent. of barium sulphate or Fuller's earth. This is rapidly removed in the centrifugal, which needs to be cleaned out every two hours or so.

In cases where the bowls can be run continuously for three or four hours, one man can work, say, five machines. Removing the dirty bowl and replacing it by a clean one takes about five minutes, while the cleaning of the bowl can be accomplished at leisure. It will contain some 10 to 15 lb. of sediment, and will take from 10 to 20 minutes to clean out. The power taken is about 1 kilowatt per hour, or about 200 lb. of steam per hour in the turbine-driven machine.

A 2 in. diameter laboratory machine of this type runs at 40,000 r.p.m., giving an effect of approximately 45,000 units. Much interesting work can be done with such a machine, such as handling serums, separating bacteria from suspensions, etc.

In conclusion, the author's best thanks are due to all those who have been good enough to assist in any way in the production of this paper.

ADSORBENTS

A meeting of the Chemical Engineering Group of the Society of Chemical Industry was held at the Engineers Club, Coventry Street, London, on Friday, March 7, 1924. Mr. J. A. REAVELL, Chairman of the Group, presiding.

The following papers were read :—

Activated Carbon. By Major V. F. Gloag, M.I.Chem.E.

Silica Gel as an Industrial Adsorbent. By Prof. E. C. Williams, M.Sc., F.I.C., M.I.Chem.E.

ACTIVATED CARBON
By Major V. F. GLOAG, M.I.Chem.E.

It is not my intention in reading this paper to give you a summary of the facts and data that have been published by various authorities on activated carbon on the Continent and in America, but to describe and show you by means of photographs and diagrams a few typical instances of its application to the recovery of solvents on works scale and generally the lines of its future development.

In doing this, however, there will be very many points not alluded to that will be of interest to you, and I trust that you will raise them in the discussion after the paper.

Before, however, starting the description of the plants the following remarks as to what is actually being produced and done in Europe to-day, using the Bayer Activated Carbon Process, may be of interest.

SOLVENT RECOVERY

Up to date plants have been built for the recovery of 8500 tons of solvent per annum, but as a considerable number of these plants have only come into operation during the last few months, only 1800 tons of solvent were recovered in 1923. In this figure is included 900 tons of benzol and 500 tons of alcohol, the other solvents recovered being ether, acetone, benzene, trichlorethylene and pyridene.

BENZOL RECOVERY IN GASWORKS

It was only in 1923 when the experimental work had been finalised at Leverkusen that plants were erected for the recovery of benzol at gasworks, and the actual capacity at work and in course of construction amounts to 1900 tons of benzol per annum.

GASOLINE RECOVERY FROM NATURAL GAS

The extended application of activated carbon for this purpose in America is well known, but actually in Europe there are at the present time plants at work and in course of construction of approximately 5000 tons of gasoline per annum, and during 1923 1050 tons of gasoline were recovered from natural gas by the Bayer process.

These figures undoubtedly show the rapid strides that the application of activated carbon to industry is taking.

In the description of plants which will follow I propose to deal with them in the following order :—

(1) The recovery of benzol in gasworks after the gas has been purified by means of bog ore.

(2) The recovery of solvents from solvent/air mixtures in works where solvents are used during the process.

(3) A description of the plant at Hüls which will shortly be put into operation for the extraction of benzol from coke-oven gas, together with the extraction of sulphur.

In the description of these plants various grades of Bayer activated carbon will be mentioned : perhaps, therefore, it would be as well for me to enumerate some of these so that during the description they can be referred to under their trade name. In case it also may interest members I have small samples of these carbons with me, together with a sample of benzol obtained at the Bernau plant on a normal run.

ACTIVATED CARBON " H "

This was the original activated carbon which was developed during the war from the decolorising carbon and is used for solvent extraction at the present time.

ACTIVATED CARBON " T "

Is an improvement and development from the " H " carbon, having a higher adsorptive capacity and greater density. This carbon is being used for all new plants for solvent extraction.

ACTIVATED CARBON " F "

Is that specially prepared for the extraction of sulphur from coal gas or coke-oven gas.

ACTIVATED CARBON " B "

Possesses a certain degree of activity, but is not active to high volatile solvents, and is used in a pre-filter for the elimination of tar fog and naphthalene, etc., from coal gas.

ACTIVATED CARBON " E "

Is the decolorising carbon, and does not enter into the scope of this paper.

Coming to the recovery of benzol in gasworks practice, that is, after the gas been purified through bog ore, Fig. 1 shows the general run of all plants.

The gas goes through the usual course in the carbonising plant, gas cooler, tar extraction plant, ammonia recovery, oxide purification and then to the activated carbon plant, from thence to meters and holders.

Plants are in operation at Bernau, Insterburg, Dessau, Leverkusen, Grenoble and one in course of construction at Flonheim.
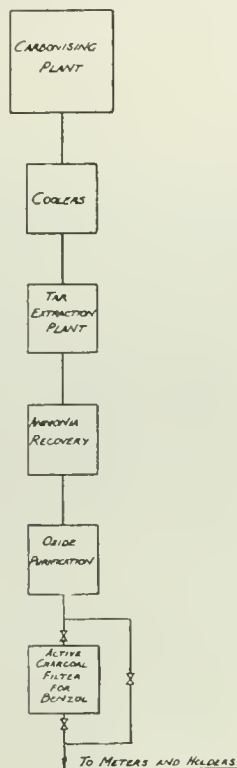
Fig. 1.

The Leverkusen plant is the plant on which the experimental work was carried out, and a photograph of this is shown in Fig. 2.

Unfortunately, a better photograph could not be obtained, but it clearly shows the two filters, benzol

1 metre diameter and 1 metre depth each containing approximately 250 kg. of " T " carbon. This plant has been fully dealt with in Dr. Engelhardt's paper on the " Recovery of Benzol from Illuminating Gas," so that I do not propose to waste time in describing it, but will later refer to this plant, giving the modifications that have been made to bring
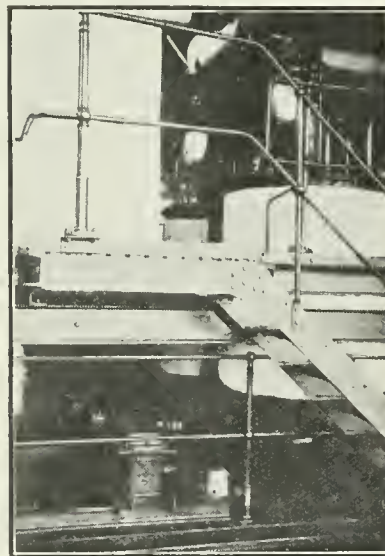
Fig. 2.

the steam consumption for this process still lower. However, it may be well to mention that after the oxide purification there is a pre-filter packed with " B " carbon which arrests any naphthalene and tar fog that may have passed the oxide purifiers. This is renewed every 8 weeks and the naphthalene is recovered by heating the carbon in retorts, the
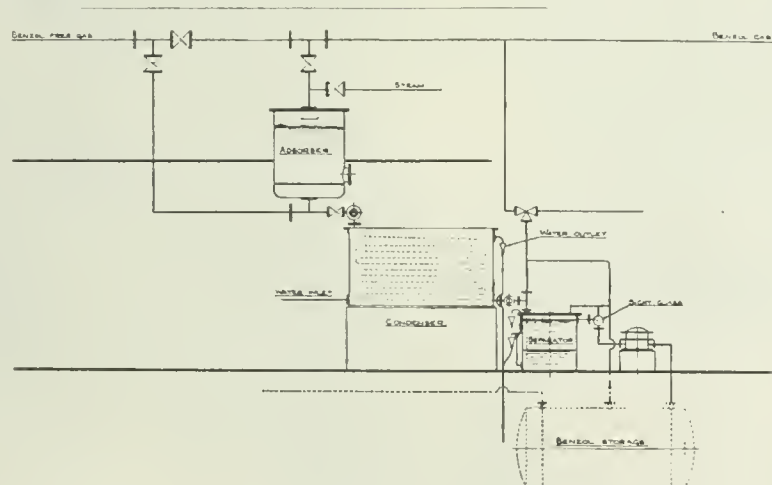
Fig. 3.

condenser and gas inlet and outlet pipes, together with the thermometers for controlling the temperatures, etc.

The capacity of this plant is about 2000 to 4000 cb. m. of gas per day, and consists of two filters

carbon being reactivated by carbonising to a temperature of 900° C.

There is a loss of carbon in this process, but it is now proposed when dealing with large quantities

of material to wash out the naphthalene with benzol and to steam out to remove this benzol so cutting out this loss.

Since the modification of the steaming arrangements was made, which was some six months ago, about 100 absorption and steaming periods have

filter is put on. Further on plants such as this where you have more than one filter you get a bigger saturation of the carbon, as you can pass gas through the filter after the " break point," placing in series another filter to act as a catch filter, but pressure conditions rule this factor.
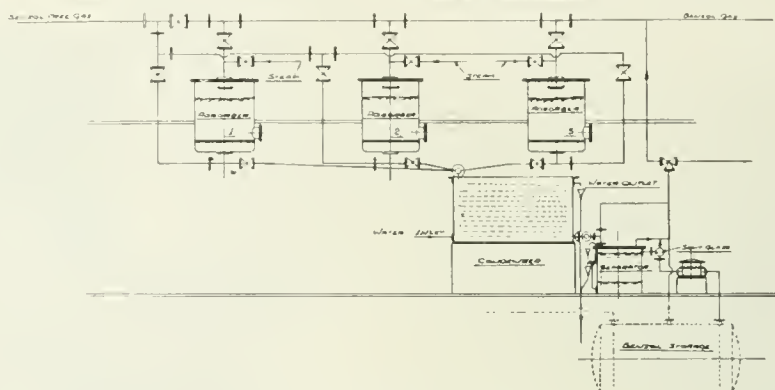
Fig. 4.

taken place, and there is no evidence that the carbon has lost any activity.

With regard to the quality of benzol recovered this will probably be raised by members in the discussion and can be then dealt with.

The plants at Bernau and Flonheim are of the one filter type as shown in Fig 3, and have a capacity of a minimum of 2000 cb. m. of coal gas per 24 hours. The filter is 1 metre dia. by 1 metre depth, and is charged with " T " carbon.

The adsorptive capacity of each absorber is some 60 kg. per day.

When the filter is being steamed out and reactivated, a process which takes $1\frac{1}{4}$ to $1\frac{1}{2}$ hours, the gas is by-passed.

This filter is fitted with coils which will be shown later in a slide showing a test plant, and on steaming indirect steam is first used to bring the temperature of the coal to a predetermined figure, and then direct steam for complete denudation.

The benzol and water driven out are condensed in an ordinary serpentine condenser and passed through a separator to the storage.

It is interesting to note that these plants are all arranged so that the vapours from the storage tanks, benzol separators, sight glasses, etc., can all be carried back to the gas stream so eliminating possible loss of benzol.

The plants at Insterburg and Grenoble are of the three filter type as shown in Fig 4, each coal filter being 1·2 metres dia. 1 metre depth of coal.

The quantity of gas capable of being treated is some 12,000 cb. m. per 24 hours, the benzol capacity being 360 kg. per day.

At Grenoble, however, the depth of coal is 1·2 metres.

Although arrangements here are made to by-pass the gas should it be necessary, actually this is not done, as when a filter has taken up the requisite quantity of benzol as shown by the flame test another

On benzol plants there is no need to dry the filter out or cool by air as with correct steam conditions, that is obtaining a temperature of 115° to 120° in the filter, the coal will only contain 5 per cent. to 10 per cent. of moisture, which is not injurious to
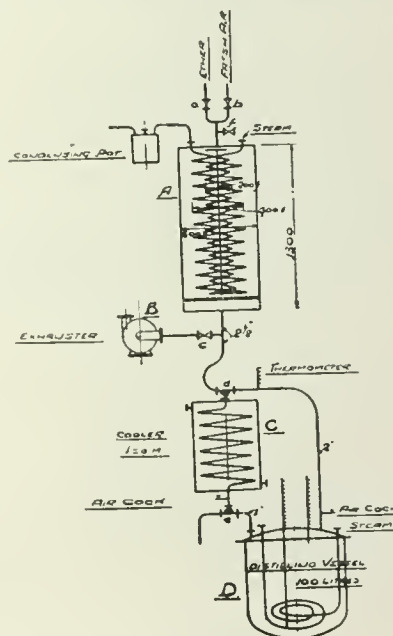
Fig. 5.

benzol recovery, and the coal cooled by means of debenzolised gas.

Fig. No. 5 shows a test plant for the extraction of solvents, etc., but before describing this I wish to mention the indirect heating of the filter.

You will notice in the filter itself which is 1·3 metres high by 0·6 metres dia., that there is a series of steam coils placed therein. These coils vary in size naturally depending upon the size of the filter,

but they are so placed that the maximum distance between any coil is 10 cm.

The use of indirect steam does away with the question of superheating and shows a considerable saving. The benzol recovery plant at Leverkusen when operating on moderate steam conditions gives under 4 kg. of steam per kg. of benzol, but if the conditions are good, that is about 5 atms. pressure,

Fig. 6.

a figure of about 3 kg. of steam is normal. There are, of course, various factors which govern the steam consumption, these being concentration of gas, degree of saturation in the filter, etc.

Turning to this small portable plant the whole is fixed in a stand and can be taken from place to place for testing purposes. The carbon filter as previously mentioned is 0·6 metre dia. and 1 metre depth of carbon, giving an approximate conte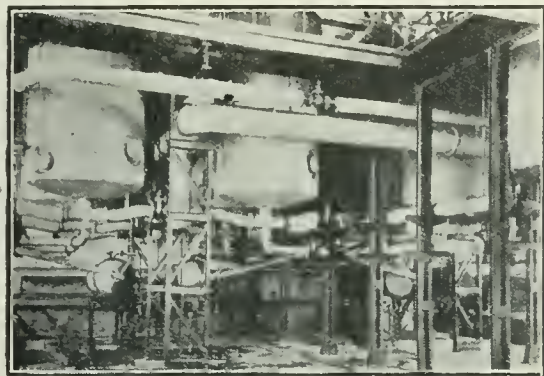nt of 250 litres of carbon. This contains an indirect heater of about 1·5 to 2 sq. metres heating surface, the heating tubes being separated by a maximum of 10 cm. The filter is fitted with the usual thermometers, gas inlets and outlets. There is a small exhauster or blower which is used to draw the gas through the filter. An ordinary cooler of 1 sq. metre surface is interposed between this and the receiving vessel and this latter vessel also acts as a still.

When distilling, the cooler which acts as the condenser for the extracted solvent from the coal is used to condense the distillate from the still. The capacity of this apparatus which is only for test and demonstration purposes, is approximately 8 kg. of ether from air containing 10 to 20 grammes of ether per cb. m., approximately 10 kg. of alcohol from air containing 20 grammes of alcohol per cb. m., 14 kg. of motor benzol from purified illuminating gas, 15 kg. of benzol from discharged air from spreading machines and dip houses in Rubber Goods Factories, approximately 15 kg. of gasoline from natural gas containing 160 grammes of vapour per cb. m.

The next three slides are photographs of an alcohol recovery plant extracting alcohol from alcohol/air mixture containing approximately 20 grammes of alcohol per cb. m.

There are 14 filters in this plant of 6 cb. m. capacity.

The plant is designed for the recovery of up to 7000 kg. of alcohol per 24 hours, but at the present moment is working at less than half this capacity owing to trade conditions.

Fig. 6 shows a view of 6 or 7 of the filters from the bottom and also shows the four alcohol condensers from which is obtained a 40 per cent. alcohol solution. This is sent to the rectification plant for treatment.

Fig. 7 shows a closer view of these filters, and the arrangement on the top platform.

The method of operation is to pass the gas containing the alcohol up through the filter, but in later plants they are mostly arranged for the gas to pass down the filter. The reason for this is that in the upward flow the carbon tends to " dance " whereas in a downward flow this does not occur.

The alcohol is extracted and the filter steamed out by direct steam at a pressure before expansion of 5 kg. per sq. cm. with a temperature of 130° C.

During the adsorption period the temperature rise of the carbon is some 20° to 30°. The filter is steamed out until the temperature at the bottom reaches 110° C., and the carbon is then dried out with hot air until a temperature of about 70° C. is reached. The filter is then blown cold with cold air and is ready to treat more gas.

The time cycle in operation when I visited the plant was as follows :—

Adsorption period, 8 hours.

Steaming, drying and cooling, 4 hours.

But this, of course, depends on factors such as concentration of the solvent/air mixture. This plant
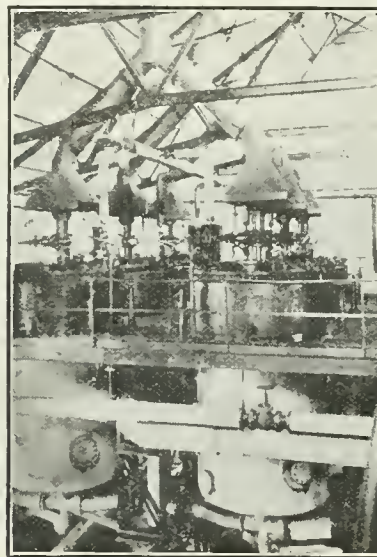
Fig. 7.

has been in operation for over 15 months, although it was erected in 1919, and no deterioration in the activity of the carbon has been noticed up to date.

The carbon used here is the " H " carbon.

Tests show that over 95 per cent. of the alcohol going to the plant has been recovered in the form of absolute alcohol during this period.

Fig. 8 shows another view of this plant.

I was unfortunately unable to get photographs

of the very fine plant in operation at Friedr. Bayer's Cellite Works at Elberfeld, where benzol and traces of alcohol are being extracted from a solvent/air mixture.

Fig. 8.

This plant has been in operation for over 15 months and has given very excellent results.

The full capacity of the plant is 1·2 kg. of solvent per minute and a filter at this capacity is saturated in 135 minutes, but owing to trade conditions actually it was being worked at less than half its load.

The carbon used here is the original " H " carbon as the plant was erected some time ago, but was not put into operation. The filters are the same size as the plant previously described, that is, 6 cb. m., each containing approximately 1700 kilos. of carbon.

The solvent air mixture varies between 10 to 30 grammes per cb. m., which affects the cycle of operation so that nothing definite can be said on this point.
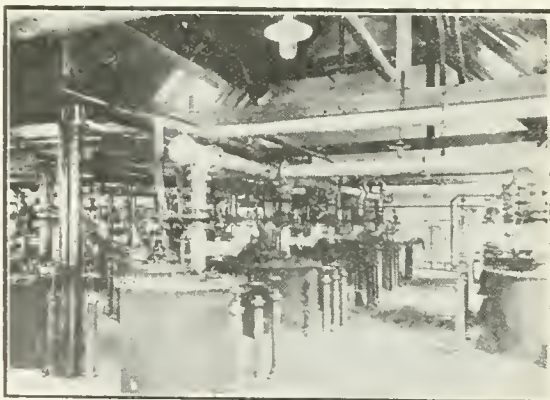
Fig. 9.

Steam, temperature and air conditions are the same for this plant as for the alcohol plant previously described, although here with the higher solvent content in the gas, the temperature of adsorption shows about 50° C. in the carbon.

When these plants were first installed there was the question of fire risk and the fire striking back into the plant, so that in all these plants the steel vessels are carefully earthed to stop any electrical effect, and further small steel vessels are placed in

the inlet and outlet mains to the filter plant filled with small pebbles to act as a fire guard. Actually as was thought at the time, these have proved unnecessary, no signs of firing has ever taken place.

With regard to the solvent recovery from solvent/air mixtures in rubber works, unfortunately here again I could not obtain photographs of the plant owing to its position, but the one seen was fitted with two filters 3 metres high by 3 metres diameter, each containing 12 cb. m. of " H " carbon. The
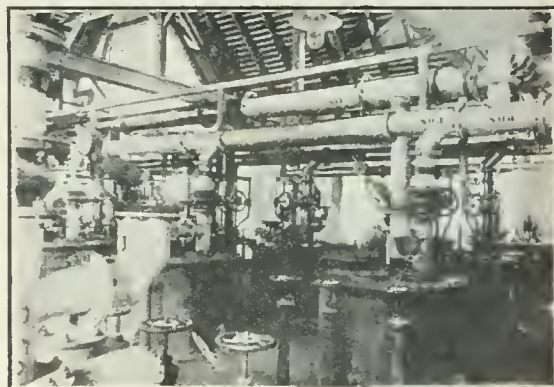
Fig. 10.

capacity of each filter on a 13 to 15 grammes solvent/air mixture is between 700 to 900 kg., but owing to the condition of the rubber industry in Germany the plant, which has been operating for the last 9 months, has only treated and recovered about 21,000 kg. of benzol.

The steaming period is 3 hours and the drying and cooling period 5 hours, which would give 2 cycles per day on full load, or have a plant capacity of between 2800 to 3600 kilos per day.

One of the biggest plants that was erected in Germany was at the Powder Factory at Haslock for the recovery of ether and alcohol. This plant has had to be demolished, but the photographs may be of interest.

Fig. 9 is a view of some of the filters.
Fig. 10 is a closer view of these filters.
Fig. 11 is a view taken underneath the filters.
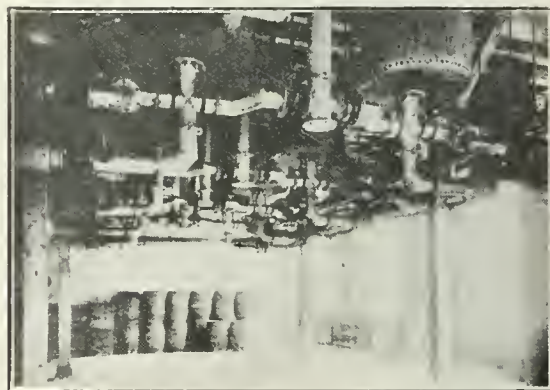
Fig. 11.

I am unable to give you any definite figures as to capacity of this plant as they are not available, but the plant was highly successful.

I will now turn to one of the most interesting developments in the application of activated carbon and that is the extraction of H_2S from coke-oven gas, which is equally applicable, of course, to the extraction of H_2S in gasworks practice.

The experimental work on this process has been carried out by Dr. Engelhardt at the Leverkusen Factory, and a paper was published on this in 1921.

On the results obtained at this experimental plant, which was erected in conjunction with the benzol plant (a photograph of which has been already shown), a full sized plant was erected at Augaste Viktoria coke oven plant to deal with 100,000 cb. m. of gas per 24 hours, with a possibility of dealing with 300,000 cb. m.

This plant would have been finished and in operation many months ago if it had not been for the shutting down of the coke ovens due to the occupation of the Ruhr, but I am pleased to say when I was at Hüls in January one of the batteries of 60 ovens was being heated up, and I am waiting

Fig. 12.

daily for information when they have been put in full operation and the process fully proved out on large scale.

Fig. 12 shows the building erected to house the plant for the recovery of benzol and sulphur and gives some idea of the size of the installation. This was designed so that it can be easily duplicated by another bay.

Fig. 13 gives a view down the house showing the benzol filters, sulphur filters and pre-filters in the course of construction.

The sulphur filters are 3 metres diameter, with a carbon depth of 1·5 metres, " F " carbon being used.

The benzol filters are 3 metres diameter by 2 metres depth of carbon, " T " carbon being charged, whereas the pre-filter which is seen in the distance, in order to give a small resistance, has only 0·6 metre depth of " B " carbon with a diameter of 4·2 metres.

The benzol filters are arranged in accordance with Slide 4 as already shown. The arrangement of the sulphur filters and sulphur extraction will be described later.

The arrangement of piping is such that all the gas passes through the pre-filter first and can then pass

through the sulphur filters and so to the benzol filters, or they can go to the benzol filters direct and then if necessary back to the sulphur filters. Further, all filters can be operated in series or
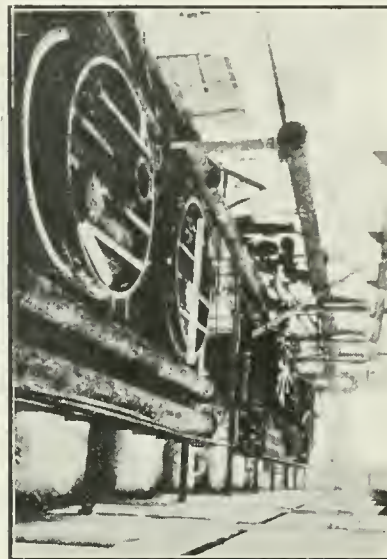
Fig. 13.

parallel. This is a somewhat expensive arrangement, but has been done with the purpose of being able to work the plant in every way possible and so obtain the fullest data on a large scale production.

Fig. 14 shows a close view of a benzol filter in course of erection.

I do not wish to explain at length the benzol

Fig. 14.

recovery, as this is the same as Gasworks practice already in successful operation, so long as we get the gas free from such bodies as naphthalene, tar fog and H_2S.

As you know coke oven gas and coal gas contain a certain percentage of oxygen and traces of ammonia, and when these, together with H_2S, come in contact with activated carbon, given the correct partial pressure and conditions, etc., the carbon appears to act as a catalyst and brings about the complete deposition of all the sulphur as such that is present

in the gas in the form of H_2S. This has been studied very carefully by Dr. Engelhardt and Messrs. Friedr. Bayer und Co., hence the development of activated carbon " F."

Fig. 15 shows diagrammatically the arrangement sulphur extraction which will be in operation at Hüls, but for clearness only two filters have been shown instead of three, which are installed.

The crude gas containing sulphuretted hydrogen after passing the pre-filter is fed with the requisite amount of air and ammonia by means of blowers to bring it up to approximately 1 per cent. of oxygen, and 0·5 gramme of ammonia per cb. m. dependent on the H_2S concentration in the gas. The gas passes through the filters, A1 or A2, where it is completely freed from H_2S with the formation of sulphur in the " F " Carbon. When one filter is saturated with sulphur to such an extent that sulphuretted hydrogen shows itself after the filter the stream of gas is switched over on to the other, and A1 regenerated. Actually at Hüls with three filters, two may be probably worked in series, one being regenerated, so that no
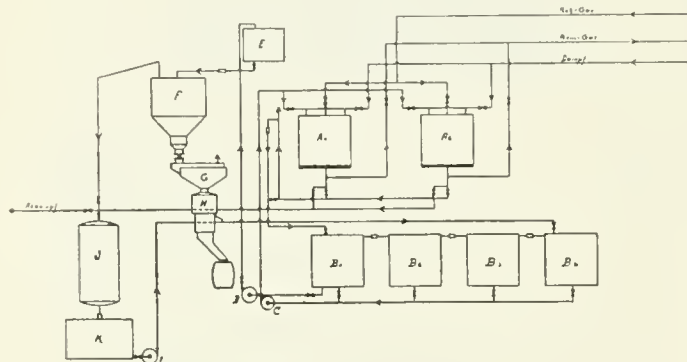
Fig. 15

trace of H_2S can get away, that is, take A1 as being in course of regeneration, the gas will be passing through A2 and A3, the gas test being taken between A2 and A3.

The regeneration of the " F " carbon has been done by two methods, the one shown on the slide is by means of ammonium sulphide.

Receivers B1 to B4 contain ammonium sulphide solution in graded strengths of polysulphide.

Taking A1 filter as being regenerated the first wash is done by means of ammonium sulphide that has already treated the sulphur saturated carbon three times, and is therefore practically saturated with polysulphide. The liquid is picked up by Pump C and delivered into the filter, a visible overflow being fitted to see when this is full. It is then run back into B1 tank and pumped by pump, D, to receiver, E, for treatment. The filter is then washed with a second wash from B2, then B3 and 4, the latter being pure ammonium sulphide. The filter, after these four washes is free from sulphur, and also from such bodies that are formed during the adsorption, such as ammonium sulphate, thiosulphate, sulphite, etc.

The filter is then blown out with direct steam, which drives out the ammonium sulphide. This is led through pipes as shown to the condenser scrubber,

J, where the ammonium sulphide is absorbed and delivered to receiver or tank, K.

This steaming is the final activation of the carbon, given correct steam conditions, and the filter can be put into the gas circuit once more to extract further sulphur.

The ammonium polysulphide solution is run from tank, E, into the sulphur boiler, F, where it is steam heated, driving out the ammonium sulphide, which is condensed and recovered in condenser scrubber, J, and collected in receiver, K.

The deposited sulphur and liquid in F is run into agitator, G, which in turn delivers in centrifuge, H, the sulphur being dropped out into suitable receivers. The liquid from the centrifuge will contain the various salts of ammonia and until this gets absolutely fouled it will be used in the condenser scrubber, J, for the absorption of the ammonium sulphide, but when fouled with thiosulphates, sulphite and sulphate it will be sent to the ammonia plant for treatment.

At Hüls, however, for experimental purposes they have further arranged for the extraction by chlorbenzol. The plant is very similar with the exception, of course, of the ammonium sulphide recovery plant.

The process depends upon the fact that chlorbenzol at a temperature of 100° C. will hold in solution 50 per cent. of sulphur, whereas at 15° C. only 4 per cent. of sulphur. Working on this at Leverkusen it has been found that with four graded washes of chlorbenzol at 100° C. the sulphur is completely extracted from the filter. This is then cooled and the sulphur crystallised out. The sulphur so recovered is freed from chlorbenzol by boiling with water, whilst the chlorbenzol adhering to the carbon in the filter is removed and recovered by steam.

In this process, however, the chlorbenzol will not remove the sulphates, sulphites and thiosulphates that may be formed, and which will be detrimental to the activity of the " B " carbon if allowed to remain. Therefore, when this process is in operation it will probably be necessary to give a periodical wash with water to the carbon for the extraction of these.

This process that I have just described is already in operation in Germany at the works of the Badischen Anilin- und Soda-Fabrik.

At Oppau a plant has been erected to deal with 1,000,000 cb. m. of water gas per day, and at Merseberg 2,400,000 cb. m. of water gas per day. These plants are only partially in operation, but are giving excellent results.

I am very sorry that I cannot give you full operating details of the plant at Hüls, but I hope to be able to do so at an early date.

I am optimistic enough to think that the results obtained at the experimental plant at Leverkusen will be fully confirmed on this big plant of a capacity of 100,000 cb. m., and if this is so it will certainly revolutionise benzol recovery in coke oven work, and give another by-product in the form of pure sulphur, and also be of intense interest to the gas world generally, and affect their purification schemes.

The rapid reaction and the small amount of material required makes the plant compare very favourably indeed with oxide purifiers.

SILICA GEL AS AN INDUSTRIAL ADSORBENT

By Prof. E. C. WILLIAMS, M.Sc., F.I.C., M.I.Chem.E.

A considerable amount of attention has been drawn in this country during the past year to the industrial use of silica gel as an adsorbent for condensable vapours from gases and as a refining agent for liquids, in particular for the refining of petroleum oils. It is proposed to deal mainly with the possibilities of its employment for the recovery of the benzene hydrocarbons from coal gas or coke-oven gas for use as a motor spirit. In view of the complex nature of coke-oven gas and the impurities which it contains, this is probably one of the most severe tests to which any solid adsorbent process can be subjected.

The work reported in this paper was carried out on behalf of the Joint Research Committee of the National Benzole Association and the University of Leeds, and it is by the permission and courtesy of that Committee and of the Executive of the Association that the results obtained are now made public. At the time when this work was commenced some two years ago, proposals had already been made in America for utilising the known adsorptive properties of silica gel for industrial purposes and patents for its manufacture and use had been taken out in the names of Patrick and others. We had no evidence, however, that any successful industrial process had been worked out or indeed that the problems of benzol recovery had been investigated at all. It was decided to investigate the problem from the beginning, and the conclusions arrived at are therefore independent of any claims put forward by the American Corporation, who are now developing the industrial applications of this substance.

As the use of inorganic gels is somewhat novel in industrial practice, a short review of their mode of preparation and properties may be desirable. It is well known that when certain hydroxides are precipitated, flocculent or gelatinous precipitates are obtained. Copper, iron, and aluminium hydroxides are typical examples, as also is silicic acid when precipitated from solutions of soluble silicates by mineral acid. Graham first showed in 1861 that by mixing with vigorous agitation a 10% solution of sodium silicate with a 10% solution of hydrochloric acid, the acid being in excess, it was possible to obtain a clear colloidal solution of silicic acid from which the dissolved electrolytes could be almost completely removed by dialysis. The silicic acid immediately after formation probably forms a true solution : it is able to diffuse through a parchment membrane and shows vapour pressure lowering and osmotic pressure. Within a few minutes,

however, the molecules of silicic acid coalesce to form larger aggregates which exhibit the properties of a true hydrosol. Graham observed that such a solution, on standing, coagulated to a firm, elastic jelly which, on drying in a desiccator over sulphuric acid, gave a hard glassy mass of great lustre. Little further work was done until the years 1890 to 1900, when van Bemmelen, in a series of studies upon the vapour pressure relationships of various hydrated oxides, made an exhaustive examination of the vapour pressure phenomena displayed by silica gel and observed the physical changes which take place in the gel during the process of drying. He showed that, on drying, the hydrogel contracted to a hard glassy porous mass which was able to absorb water and other vapours. He investigated particularly the effect of the concentration of liquid from which the gel was prepared and the time taken for the drying of the gel, upon its porosity and adsorptive power (Z. anorg. Chem., 1897, **13**, 239 ; 1898, **18**, 117). Zsigmondy, Bütschli, Bachmann, Anderson and Patrick have also investigated the structure of silica gel by ultra-microscopical methods or by the determination of vapour pressures of liquids adsorbed by the gel.

The fact that a jelly containing as many as 100 molecules of water to each molecule of SiO_2 is a moderately stiff elastic structure indicates some strong bond between the silica and water. Cases of two liquids forming an apparently solid structure, *e.g.*, butter : or a liquid and gas, *e.g.*, whipped cream or white of egg, are well known and may be explained on the basis of a cellular or honeycomb structure. Silica gel is unlike these in that the water in the gel can be completely replaced by other liquids or may be completely removed without affecting the characteristic structure of the gel. These properties preclude the possibility of a honeycomb or cell-like structure, and direct ultra-microscopical examination has failed to establish definitely what the true structure is. It may conceivably consist of threads of silica particles intertwining indiscriminately and in all directions after the fashion of the threads in a ball of cotton wool : or may, as Zsigmondy represents it, be simply a formless collection of particles of silica held together by the adsorption of water at their surfaces. Either of these views would account for the open structure which, as will be seen, the gel must possess.

Van Bemmelen (Z. anorg. Chem., 1898, **18**, 140) investigated the physical changes which occur when silicic acid jelly is gradually deprived of water

Table I. (reproduced from his paper) records the volume and specific gravity changes during dehydration. The last column shows the calculated specific gravity of a jelly containing the proportions of water and silica indicated in the first column, whilst the third column shows the specific gravities actually observed.

TABLE I.

Mols. H₂O per mol. SiO₂.		Volume of gel		Specific gravity.		
				Observed		Calculated
122	..	29	..	1·00	..	1·01
75·7	..	18	..	1·02	..	1·02
45·2	..	11	..	1·04	..	1·04
23·2	..	4	..	1·08	..	1·09
11·3	..	3	..	1·14	..	1·14
2·8	..	1	..	1·40	..	1·41
2·2	..	0·86	..	1·51	..	1·49
1·7	..	0·75	..	1·58	..	1·56

Transition point

1·00	..	0·73	..	1·40	..	1·72
0·39	..	0·73	..	1·21	..	1·93
0·30	..	0·73	..	1·17	..	2·00

It will be seen from column 2 that the gel rapidly contracts as water is removed, until a point is reached at which the water content is some two molecules per molecule of SiO₂. Up to this point the decrease in volume of the gel exactly corresponds to the volume of water removed, as is shown by the agreement between the observed and calculated specific gravities. The particles of silica have, therefore, gradually closed together while the spaces between them have remained completely filled with water. After this point, however, dehydration does not cause further contraction. The silica particles have assumed a final rigid structure and further dehydration leads to the driving out of the water from between the silica particles, the spaces being filled with air as rapidly as the water is removed. With the first entry of air the gel loses its transparency and becomes chalky white in appearance, due to the refraction and reflection of light from the minute air bubbles, breaking the continuity of the water within the gel. As dehydration proceeds still further practically the whole of the water is removed and the interspaces become filled with air and the gel regains its transparency. The gel in this final form is a hard, clear, lustrous substance, to all appearances like fragments of broken glass, and it is in this form that it is employed as an adsorbent.

It is not necessary to enter more deeply into the fascinating studies which have been made upon the structure of silica and other gels. The essential point is that, by the formation of the hydrated gel, silica is obtained in a state of extreme subdivision widely dispersed in a water medium, the particles being held in a more or less rigid structure. On dehydration, either in a desiccator, or by heating, the gel contracts, the particles close together until finally they can contract no further. The interspacial water is then driven off, leaving a highly porous, but optically homogeneous, substance. The determination of the actual size of the pores is a matter of some difficulty. The fact that they are beyond the resolving power of the ultramicroscope indicates a diameter of the order of some 10 μμ, while determinations made by Anderson (Z. physik.

Chem., 1914, 88, 191) of the vapour pressures of liquids held in the pores lead to a value of 2·5 to 5·5 μμ. Anderson's method is, however, open to some criticism in so far as his calculations are based on the assumption that the vapour pressure of liquids in such minute pores obeys the same laws as apply to liquids in bulk.

Van Bemmelen (Z. anorg. Chem., 1897, 13, 239) further investigated the influence on the porosity and adsorbing power of silica gel, of variation of such conditions as the concentration of liquid from which the gel was coagulated, and the time taken from the drying of the gel. Either of these factors would tend to affect the size and nature of the silica aggregates formed, and therefore the structure and porosity of the dried gel.

The nature of adsorption by porous bodies.

Almost every solid surface, whether porous or otherwise, is able to adsorb small amounts of gas or vapour. The extraordinary difficulty, for example, of removing the last traces of moisture from the surface of a glass vessel is well known. The amount of vapour adsorbed by a given weight of substance must clearly be greatly enhanced when the substance exposes so vast a surface as does the silica in silica gel. (The surface of a cube of 1 cm. edge is 6 sq. cm., but if that cube were broken up into cubes of 10 μμ edge, the surface exposed would be 600 sq. metres, or some 6500 sq. ft.) This type of adsorption is a purely surface effect, and is dependent upon definite forces of attraction between the molecules of the solid and the molecules of the gas or vapour. These forces are generally assumed to be of a chemical or polar origin and lead to a definite orientation of the adsorbed molecules at the surface of the solid. It is therefore to be expected that adsorption of this type will be affected by the molecular arrangement of the vapour molecules or of the molecules of the adsorbent, and that adsorption will be largely selective. Such adsorption is met with particularly in the case of certain forms of active charcoal, and is in many cases a very desirable property from the industrial standpoint.

A second type of adsorption, which may come into play when a highly porous adsorbent is used, is purely physical in nature. When a capillary glass tube is dipped into water, or other liquid which wets the surface of the glass, the water rises in the capillary to a height dependent upon the radius of the tube and the surface tension of the liquid. The vapour pressure of the liquid over the meniscus in the tube is less than that over the same liquid contained in an open vessel and for any diameter of the tube, the vapour pressure of the liquid contained in it may be calculated. (The reverse calculation is the basis of Anderson's determination of the pore diameter already mentioned.) It is evident that if a sufficiently fine capillary tube is brought into an atmosphere of condensable vapour in which the vapour pressure of the vapour is higher than that which would be in equilibrium with the liquid contained in the capillary, then a partial condensation of the vapour in the capillary will take place. Silica gel may be regarded as a mass of such fine capillaries.

Since these capillaries are formed by the juxtaposition of silica particles they are not of the same diameter throughout. The capillaries themselves are probably wedge-shaped and of different sizes. It is possible, therefore, to consider the pore space of any fragment of gel as forming a single long wedge-shaped capillary, which in the presence of a condensable vapour will become filled with liquid from the apex of the wedge outwards, until a point is reached at which the diameter of the capillary is such that the vapour pressure at the liquid meniscus is equal to that of the vapour in the surrounding atmosphere. The amount of vapour adsorbed is therefore a function of the vapour pressure. It is evident that it must also be a function of the temperature. Change in either vapour pressure or temperature alters what may be called the "ease of condensation" of the vapour and it is more convenient, as pointed out by Patrick, to express such ease of condensation in terms of a single condition of the vapour instead of in terms of two. This single condition, which determines the degree of adsorption, has been defined by Williams and Donnan (Trans. Faraday Soc., 1914, 167), who showed from a thermodynamic analysis, that if the ratio of the actual vapour pressure of a vapour to the saturated vapour pressure is equal to unity, the amount of vapour adsorbed is a finite maximum and is independent of temperature. Patrick has termed this ratio of partial pressure to saturated pressure, the " corresponding pressure " of a vapour, and has shown that in the case of adsorption of sulphur dioxide by silica gel the same amount of SO_2 is taken up whatever the temperature, provided only that the corresponding pressure is equal to unity. The amount of vapour adsorbed by a given mass of gel is so great that it must be regarded as condensed in the liquid state. It is therefore preferable, in comparing the adsorption of different vapours to consider adsorption in terms of liquid volume, rather than as weight or volume of vapour. Bachmann (Z. anorg. Chem., 1913, 79, 202) showed that when silica gel is exposed to the saturated vapour of water, benzene, or chloroform, the same liquid volume of each vapour is adsorbed, and furthermore that this volume is equal to that taken up when the gel is immersed in the respective liquids. The finite maximum adsorption which takes place when silica gel is exposed to a vapour at a corresponding pressure equal to unity represents therefore a complete filling of the pore spaces of the gel, and the principle of Williams and Donnan can in this case be extended to read : " that for corresponding pressures equal to unity the same liquid volume is adsorbed, whatever the vapour or whatever the temperature."

The adsorption of vapours by silica gel is regarded by many workers as a purely physical capillary phenomenon of the type described above, but there are several observations which do not agree with this point of view. It is not intended in this paper, however, to enter into the theory of the mechanism of adsorption by silica gel, but rather to deal with some of its industrial applications. For this purpose the capillary theory of adsorption presents a useful concrete picture of the mechanism by which vapours

are adsorbed, and is sufficient to explain many of the phenomena observed.

The immediate problem before the National Benzole Association was the possibility of using solid adsorbents in lieu of wash oil for the recovery of the condensable hydrocarbons from coal gas and coke-oven gas. The use of active charcoal and various inorganic gels was considered, and silica gel was chosen for first investigation because of certain definite advantages which it appeared likely to possess. Coke-oven gas contains many impurities likely to affect or destroy the adsorptive power of solid adsorbents, chief amongst which are the presence of tar fog in the form of a fine suspension ; the vapours of high-boiling hydrocarbons, or of compounds which tend to polymerise to high-boiling resinous substances on the body of the adsorbent, which would not be removed by the usual processes of distillation ; and sulphur compounds, including hydrogen sulphide, which might act upon the material of the adsorbent. The possibility of contamination with resinous organic matter demands that the adsorbent should be able to withstand heating in an oxidising atmosphere to a temperature sufficient to burn off such organic compounds and the deposit of carbon which is formed from them in the pores of the adsorbent during the initial period of heating. Ferric hydroxide gel, a substance of similar structure to silica gel was found to be unable to resist the high temperatures involved ; it broke up completely to a finely powdered oxide which showed no adsorption capacity ; there is also the danger that such a metallic hydroxide gel, even if able to withstand high temperatures, would be acted upon by the hydrogen sulphide in the gas. Active charcoal is at a disadvantage when heating in an oxidising atmosphere to a temperature of about 600° C. is involved ; this disadvantage might be obviated by carefully purifying the gas before passing it over the charcoal. Silica gel is commendable on account of its chemical inertness and its ability to withstand high temperatures. These two factors counteract to a great extent the undoubted superiorities which active charcoal possesses, particularly its greater adsorptive power for vapours at low concentrations and its selective adsorption of hydrocarbons in preference to water vapour.

In the investigations undertaken, adsorption efficiency and capacity were measured by the dynamic method rather than by the static method adopted by van Bemmelen, Anderson and Patrick, since this method is more closely allied to conditions which would obtain in large scale practice. After carrying out these investigations we became acquainted with a paper by E. B. Miller (Chem. and Met. Eng., 1920, 23, 1155, 1219, 1251), in which the examination of mixtures of air with benzene, ether, acetone, and other solvents by the dynamic method is described and, in so far as our results cover the same ground, they are in complete agreement with Miller's.

The silica gel used was prepared by the method recommended by Patrick (J. Amer. Chem. Soc., 1920, 42, 951) and preliminary experiments were made to determine the efficiency and capacity of the gel

for the adsorption of benzene from mixtures of air and benzene.

The apparatus is shown in Fig. 1. A current of air was drawn through a calcium chloride drying tube, A, a calibrated flow-meter, B, a three-bulb saturator, C, containing benzene, the U-tube, D, containing the gel under examination, and two additional guard tubes (E, F) also containing gel and serving to determine the amount of benzene passing D. The release valve, G, ensures a steady suction through the apparatus.

was recovered in the three gel tubes. In Table II the adsorption by D only is shown. The data from this table are plotted in Fig. 2.

It will be seen that benzene is adsorbed almost quantitatively for a period of three hours, during which time the gel takes up 40% of its own weight of benzene. After three hours the efficiency of adsorption falls off rapidly to zero, the gel becoming saturated at 55·4% benzene content. (It is interesting to note that the gel used in this experiment had been heated 30 times to 600° C. for a period of

FIG. 1.

The gel tube, D, contained 12·606 grams of gel, and air was drawn through the apparatus at a rate of 100 c.c. per minute. The saturator, C, and the three U-tubes were weighed at frequent intervals, and it was found that the whole of the benzene lost from C

one hour, and its adsorptive capacity had not appreciably fallen from the value initially observed.)

TABLE II.

		Benzene in gas, 9·5%			
Time from start Mins.	Temperature during interval °C.	Total weight C₆H₆ passed Grms.	Total weight C₆H₆ adsorbed Grms.	Adsorption efficiency during interval %	Per cent. benzene in gel %
0	—	—	—	—	—
30	18·0	0·8600	0·8600	100·00	6·87
60	18·0	1·7980	1·8010	100·10	14·30
100	18·5	2·8675	2·8627	99·70	22·70
120	18·5	3·3960	3·3945	99·70	26·90
130	18·5	3·7110	3·7055	98·70	29·50
140	18·5	4·0200	4·0130	99·15	31·40
150	18·5	4·3435	4·3355	99·70	34·30
160	18·5	4·6480	4·6375	99·20	36·60
170	18·5	4·9865	4·9735	99·20	39·50
		Stoppage overnight.			
180	18·0	5·2775	5·2565	97·20	41·60
190	18·0	5·5880	5·5485	94·00	44·00
200	18·5	5·9020	5·8315	90·10	46·30
220	19·0	6·5605	5·3545	79·40	50·30
250	19·0	7·5533	6·8530	50·20	54·20
270	19·0	8·1715	6·9700	18·90	55·10
290	19·0	8·8585	6·9775	1·10	55·38

This adsorption of 55·4% of benzene was from air saturated with benzene. The efficiency of adsorption from a gas containing benzene at a concentration corresponding to that actually present in coal gas or coke-oven gas was next examined. The content of benzene in coal gas may be taken as approximately 0·8% by volume, corresponding to about 8% saturation.

The apparatus was essentially as in Fig. 1, with the exception that a second metered stream of dry air could be mixed, by means of a connexion, with
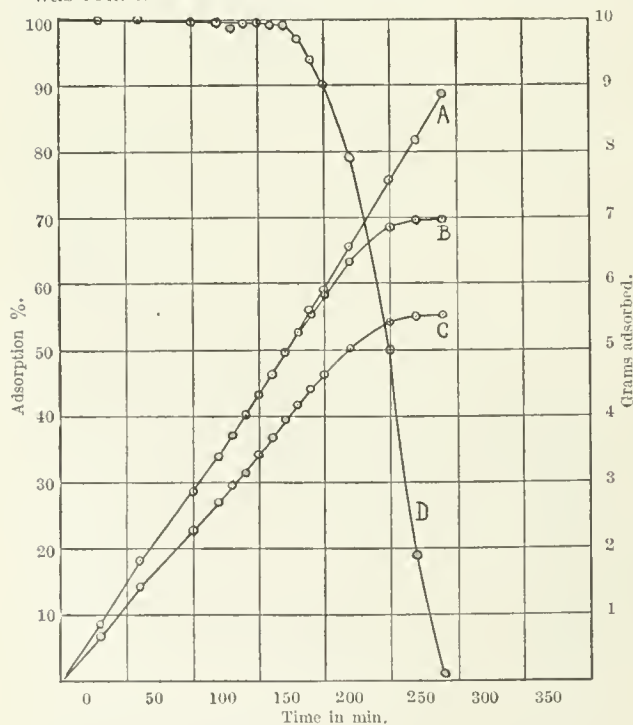
A. Weight of C₆H₆ passed.
B.   "   "   adsorbed
C.   % C₆H₆ in gel.
D.   Efficiency of adsorption %.
FIG. 2.

the stream of air saturated with benzene.   By adjusting the proportion of air added, any desired concentration of the mixed gas was obtainable.

The figures of Table III and curves in Fig. 3 show the efficiency and capacity of two gel tubes placed in series in a gas stream containing 0·84% of benzene. The first tube contained 5 grams and the second 9·5865 g. of gel ; the rate of gas stream was 182 c.c. per minute, giving a time of contact of 2 to 3 seconds in the first tube and about twice that period in the second.
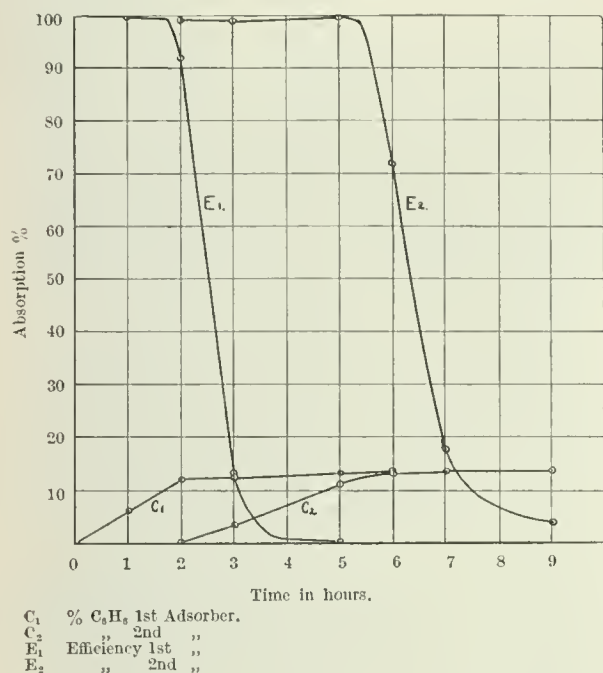
of intermediate concentration is not of immediate interest from the standpoint of recovery from coal gas, but it can readily be determined. Table IV and Fig. 4 show the saturation capacities of the gel for different concentrations of benzene at 16° C. This is the usual form of adsorption isotherm.

Concentration of benzene. Vol. %.
Dry benzene-air mixture.
Variation of benzene adsorption with its concentration. Temp.=16° C.
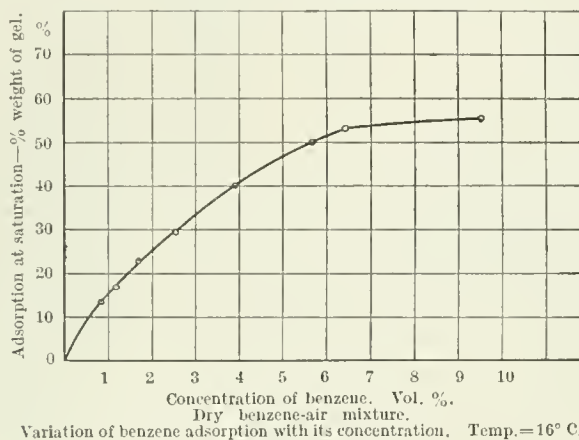
FIG. 4.

TABLE IV.

Saturation values for gel in equilibrium with benzene of different concentrations.

Average temperature, 16° C.

| Concentration benzene %	Partial pressure benzene mm. of Hg.	Per cent. weight adsorbed at saturation
0·84	6·40	13·65
1·19	9·05	16·85
1·69	12·8	22·96
2·55	19·3	29·50
3·90	29·7	40·00
5·64	43·0	50·20
6·41	48·9	53·40
9·50	72·2	55·40

C₁ % C₆H₆ 1st Adsorber.
C₂   „   2nd   „
E₁ Efficiency 1st „
E₂   „   2nd „

FIG. 3.

TABLE III.

(See curves, Fig. 3).

Adsorption of benzene from a mixture of air and benzene containing 0·84% of benzene by volume.

			Adsorption by first tube			Adsorption by second tube		
Time from start Hrs.	Average temp. during interval	Total weight of benzene passed	Total	Efficiency during interval	Benzene % in gel	Total	Efficiency during interval	Benzene % in gel
0	—	—	—	—	—	—	—	—
1	16	0·3172	0·3165	99·70	6·30	—	—	—
2	17	0·6440	0·6170	92·00	12·34	0·265	99	0·028
3	18	0·9990	0·6615	12·60	13·23	0·3340	99	3·500
5	19	1·7250	0·6630	0·27	13 26	1·0600	100	11·100
			Stoppage overnight					
6	16	2·0327	0·6897	8·7	13·79	1·2622	72·0	13·200
7	17	2·3242	0·6770	Loss	13·50	1·3160	17·7	13·700
			Stoppage over week-end					
9	15	2·9012	0·6805	0·6	13·65	1·3380	3·85	13·900

Influence of water vapour on the absorption of benzene from a benzene-air mixture by silica-gel.

The results show a saturation value of the gel at 16° C. of 13·7% by weight, while 11% is taken up at 100% efficiency. These are the adsorption values which would be expected when working on an average coal gas, provided that the adsorption were not affected by other factors.

Tables II and III show the adsorption obtainable from a gas containing 9·5% and 0·84% of benzene. The adsorption capacity of gel in gases

The above experiments were made with simple benzene-air mixtures. The case of coal gas or coke-oven gas is not so straightforward. Apart from substances which may foul the gel and so reduce its activity, there are a number of different vapours, each with its own particular partial pressure and its own corresponding pressure, p/p_0 as defined by Williams and Donian (see page 99 T).

The partial and corresponding pressures of some

of the main condensable vapours in normal coal gas are found to be as follows :—

TABLE V.

		Partial pressure (p) mm. Hg.		Saturation pressure (p⁰) mm. Hg.		Corresponding pressure p'/p'.
Pre-benzol	..	0·165	..	195	..	0·0009
Benzene ..	..	5·880	..	60	..	0·0980
Toluene ..	..	1·158	..	14	..	0·0825
Xylene ..	..	0·528	..	4	..	0·122
Naphthalene	..	0·019	..	0·03	..	0·65
Water ..	..	12·780	..	12·78	..	1·00

The figures for pre-benzol, benzene, toluene, and xylene are those given by T. F. E. Rhead (Gas J., 1917, **137**, 207) as representative of a normal coal gas at 15° C. The naphthalene figure corresponds to a content of 6 grains per 100 cubic feet ; in practice even higher figures may be obtained. The water figure is based on the assumption that the gas is saturated. All the saturation pressures are for 15° C. It is clear from the principles of capillary adsorption discussed on p. 99 T, that if the adsorption of these constituents could be considered independently, the saturation capacity of the gel would be greatest in the case of water which is present at a corresponding pressure equal to unity, and would, therefore, be taken up by the gel to the extent of approximately 60% by weight (55% of benzene was taken up from a saturated benzene vapour, and the same volume of water would be adsorbed from a saturated water vapour). Naphthalene would come next as having the next highest corresponding pressure, then xylene, benzene, toluene, and pre-benzole in rapidly decreasing amounts. This is what would occur if a gas were passed containing each of these constituents singly at the concentrations mentioned, but obviously a vastly greater volume of gas would have to be passed in the case of naphthalene than, say, in the case of benzene or water, since the actual concentration of naphthalene in the gas is so small. What, then, will happen when a gas is passed containing all these constituents together ? Examining the case where two only of the vapours are present, and choosing two which have widely different partial and corresponding pressures—benzene and naphthalene—it will be assumed for simplicity that the naphthalene is present to saturation in the gas, which is a possible condition. The corresponding pressure of naphthalene is then equal to unity, and if sufficient gas were passed, the naphthalene would be adsorbed by the gel to the extent of some 70% by weight (benzene at $p/p_0 = 1$ gave 55% ; this multiplied by the ratio of specific weights of naphthalene and benzene, 1·15/0·88, gives the weight of naphthalene adsorbed at $p/p_0 = 1$). Thus, in order to saturate 10 g. of gel with naphthalene, it would be necessary to pass 7 g. of naphthalene, which at 9·4 grains per 100 cu. ft. corresponds to 1150 cu. ft., or 32,500 litres. In the meantime, however, the gel has long reached its saturation point with respect to benzene. This point, for a partial pressure of 6 mm. at 15° C., is approximately 14% of the weight of the gel, and is, therefore, reached when 1·4 g. of benzene, or 50 litres of gas, have been passed. At this point the naphthalene passed amounts only to 0·0108 g. There

is a question now of what will take place if the gas continue to pass after the saturation point to benzene is reached. Clearly, no more benzene will be adsorbed by the gel, but will the naphthalene simply fill up the balance of pore space left to it by the benzene, or will it displace the benzene already there ? Where, as in this case, there is presumably no question of preferential surface adsorption coming into play, the answer is not self-evident, and there is no published work which gives a satisfactory reply.

In order to investigate this point, which appeared likely to be of importance in the recovery of benzol from gases, it is necessary to consider adsorption from a gas containing two condensable vapours whose separate distribution in the gel can be determined at frequent intervals. Working on a small laboratory scale it was impossible to use, say, benzene and toluene, or benzene and xylene, as the adsorbed vapour would have to be distilled out of the gel at the completion of each time interval before an analysis could be made, and as only a fraction of a gram is collected during each interval, this would be impossible.

As the adsorption by silica gel is supposedly a purely physical phenomenon and not dependent on the chemical nature of the vapour, it was not necessary to restrict attention to hydrocarbons. Sulphur dioxide or ammonia might be used as a second vapour in conjunction with benzene, as they would be easily distinguished from benzene and separately traceable in their adsorption relations. Actually, water vapour was chosen as being not only separately traceable, but also a material constituent of coal gas, and one the action of which will have to be considered. We have the following data :—

Partial pressure of benzene in gas	..	..	6·0 mm.
Partial pressure of water in gas	..	..	12·8 mm.
Volume of liquid water from 22·4 litres vapour	..	18 c.c.
Volume of liquid benzene from 22·4 litres vapour	89 c.c.

The volume of water vapour in the gas is 2·13 times that of benzene vapour ; on the other hand, a given volume of benzene vapour yields five times as much liquid volume as an equal volume of aqueous vapour. If, therefore, we express the ratio of benzene to water in the gas as volume of liquid condensates, which is the important point from the view of adsorption, we have benzene : water = 5 : 2·13 or 2·35 : 1.

This is a similar, though less extreme, case to that of benzene and naphthalene, discussed previously. The benzene, although not capable, like the water, of approaching the maximum saturation value (on account of its low corresponding pressure), is yet present in considerably greater proportion, as liquid volume, than the water, and is, therefore, able to enter the gel freely in the first stages of adsorption before sufficient water has arrived to exclude it—assuming for the moment that the vapour of the higher corresponding pressure does tend to exclude or drive out that of the lower. It is impossible to forecast what should occur in such a mixture, and experiments were carried out to test the above points.

Adsorption of mixed benzene and water vapours.

A gas containing both benzene and water vapour in known concentrations was prepared by mixing two metered streams, one having passed through a water saturator and the other through a benzene saturator, as shown in Fig. 5. Each stream was dried in a gel. The gas mixture contained 0·86% of benzene and 1·85% of water by volume with partial pressures 6·5 mm. and 14·2 mm. respectively. The temperature of the saturator and adsorption tubes was 18° C. The saturation pressure of water vapour at 18° C. is 15·46 mm. The gas was therefore not
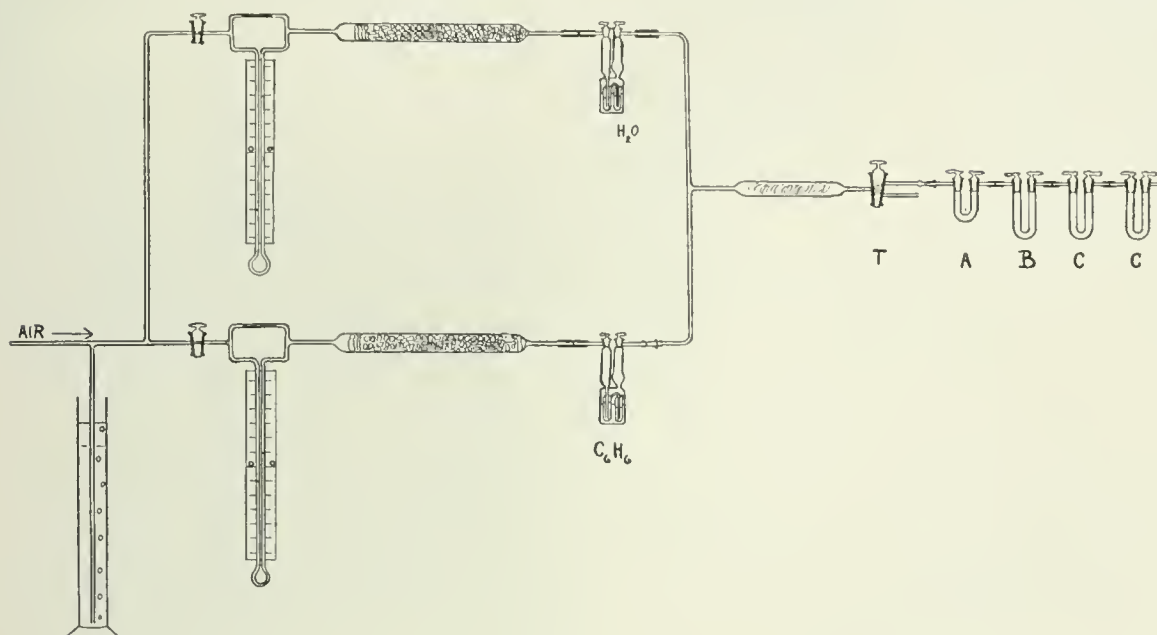

Fig. 5.

long calcium chloride tube before passing through the saturator. After mixing in an intermediate vessel, the gas passed through : A, a small tube containing 3·708 g. of gel ; B, a tube containing granular calcium chloride ; C, two guard tubes filled with silica fully saturated with water. The mixed gas was passed through the adsorption tubes for 21·7 hours at a rate of 135 c.c. per minute. By periodic weighings of the water and benzene saturators, the gel tube, A, containing the gel under examination,

TABLE VI.

(See Curves, Fig. 6).

The adsorption of benzene from a mixture of benzene and air containing water vapour.

Rate of gas stream .. .. 135·5 c.c. per minute.
Benzene in gas .. .. 0·86% by volume.
Water in gas .. .. 1·85% by volume.

Time Hrs.	Temp. ° C.	Water distribution			Benzene distribution			Total vapours in gel		Per cent. vapours in gel		Per cent. of benzene passed adsorbed by guard tube in each period.
		Passed Grms.	Adsorbed by		Passed Grms.	Adsorbed by		H_2O Grms.	C_6H_6 Grms.	H_2O Grms.	C_6H_6 Grms.	
			1st gel Grms.	CaCl₂ Grms.		1st gel Grms.	Guard Grms.					
0·0	—	—	—	—	—	—	—	—	—	—	—	—
2·0	18·0	0·1794	0·1726	0·0068	0·4395	+0·2945	0·1450	0·1726	0·2945	4·46	7·92	33
3·2	19·0	0·1128	0·0946	0·0812	0·2615	—0·0515	0·3130	0·2672	0·2430	7·20	6·56	120
4·4	19·0	0·1162	0·0800	0·0362	0·2770	—0·0270	0·3040	0·3472	0·2160	9·35	5·82	110
7·4	20·0	0·3433	0·1949	0·1484	0·7885	—0·0557	0·8442	0·5421	0·1603	14·65	4·33	107
8·9	21·5	0·1703	0·0814	0·0889	0·4053	—0·0134	0·4187	0·6235	0·1469	16·80	3·95	103
9·7	21·5	0·0885	0·0418	0·0467	0·2247	—0·0001	0·2248	0·6653	0·1468	17·98	3·95	100
13·7	20·0	0·4373	0·1853	0·2520	1·2183	—0·1140	1·3323	0·8506	0·0328	20·30	0·88	110
15·2	17·5	0·1029	0·0294	0·0735	0·2796	—0·0215	0·3011	0·8800	0·0113	23·70	0·30	108
17·7	18·5	0·2415	0·0900	0·1515	0·5361	—0·0088	0·5449	0·9700	0·0025	26·10	0·06	102
18·7	17·5	0·0796	0·0222	0·0574	0·2120	+0·0077	0·2043	0·9220	0·0102	26·70	0·27	97
19·7	18·0	0·0854	0·0127	0·0576	0·2122	+0·0031	0·2091	1·0200	0·0133	27·50	0·35	99
20·7	18·5	0·1043	0·0343	0·0700	0·2143	—0·0024	0·2437	1·0543	0·0109	28·40	0·29	100
21·7	18·5	0·1053	0·1033	0·0740	0·2460	—0·0003	0·2403	1·0856	0·0106	29·40	0·28	100

the calcium chloride tube, and the final guard tubes, a determination of the amounts of water and benzene adsorbed by A at any moment was made. The results are given in Table VI and are plotted in Fig. 6. (The determinations for the first three points of the curves in Fig. 6 are not shown in Table VI; they were obtained from an independent experiment under exactly similar conditions.)

It is evident, therefore, that the presence of water vapour is a factor to be seriously considered in the problem of recovering benzene from gas. It not only prevents the benzene attaining the normal saturation value corresponding to its vapour pressure, but also, in the event of the gas being passed over the gel in too great a volume, leads to a complete washing out of the benzene initially adsorbed, even though
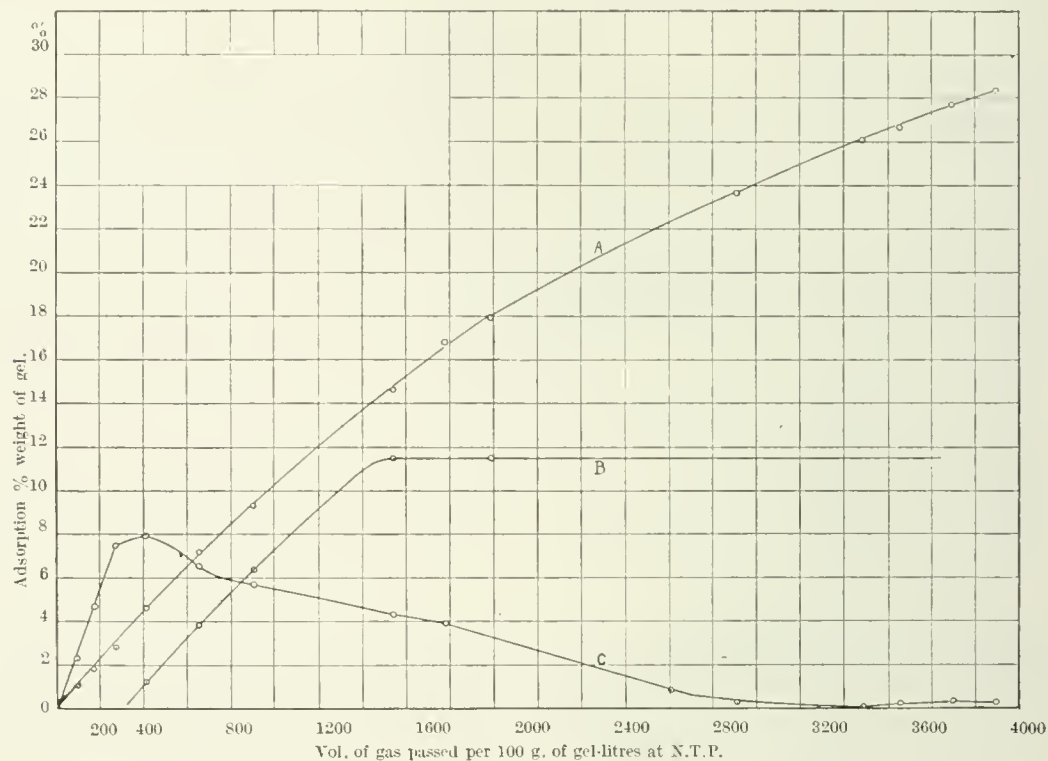
Influence of water vapour on the absorption of benzene from a benzene-air mixture by silica-gel.

Curve A = % H₂O in gel.
  „   B = % C H in guard tube (gel) after dehydrating gas.
  „   C = % C H in gel.
% Benzene in gas = 0·86%.   Temp. – 18° C.
% Water in gas = 1·85%.   Rate of gas = 7·6 litres per hr. N.T.P.

Fig. 6.

Curve A shows the adsorption of water, expressed as per cent. weight of the gel. Curve B shows the adsorption of benzene in the first gel guard tube, and is therefore a check upon the adsorption which would have been obtained had the gas not contained water vapour. Curve C shows the actual benzene adsorption from the wet gas. It will be observed that in the initial stages benzene was taken up to the extent of 7·92% of the weight of the gel, but as the passage of gas was continued, the benzene was gradually displaced by water, until finally no benzene remained in the gel. The water, on the other hand, gradually accumulated in the gel, and at the completion of the experiment was still being rapidly adsorbed. A further proof of the fact that the benzene is displaced is shown by the figures in the last column of Table VI, from which it is seen that during every interval after the first two hours, more benzene was recovered in the guard tubes than was lost from the benzene saturator.

the concentration of benzene in the gas remains constant. The explanation probably lies in the relative ease of wetting of the silica surface by water and by benzene. If the silica surface is coated preferentially by a film of water, the benzene is not able to wet the surface, and therefore depends for its retention upon its power to wet, or be adsorbed by, the water film. If benzene is adsorbed less strongly at a water face than at a silica face, then as the water film develops over the surface of the silica, benzene must be driven out, whether the adsorption is regarded as a molecular surface adsorption or as a purely capillary condensation.

The next step to be taken was the examination of the adsorption of hydrocarbons from actual coal gas. A supply of purified town gas was drawn from the mains of the Leeds gas works at a point immediately after the iron oxide purifiers. A metered stream of this gas was passed through a series of six adsorption tubes, each containing 12 grams of

silica gel, until benzene just commenced to pass the last tube. The contents of each tube were distilled *in vacuo* and collected in a graduated receiver cooled to –10° C. The quantities obtained on such a scale were too small to permit of an accurate analysis, but were sufficient to show the selective adsorption of the various constituents of the gas along a column of adsorbent. The approximate analysis is expressed in Table VII below, in terms of water, benzene, and toluene. The small amounts of hydrocarbons with boiling points higher than toluene and lower than benzene have been neglected.

TABLE VII.

Adsorption of water and hydrocarbons from coal gas in a six-section column.

No. of tube in series	Vol. water c.c.	Vol. hydro-carbons c.c.	\%$_0$ Composition of hydrocarbons C_6H_6 \%	C_7H_8 \%	Boiling point of hydro-carbons ° C.
I	1·15	1·90	25	75	98·9
II	0·65	1·40	65·5	34·5	87·7
III	0·15	1·15	100	0	80·1
IV	0·05	1·20	100	0	80·0
V	0·0	1·45*	100	0	79·7
VI	0·0	1·10	100	0	79·5

* This volume includes a drop of the distillate from the previous tube.

It is evident from these figures that a considerable degree of fractional adsorption takes place, and that the water and higher boiling hydrocarbons are concentrated at the entrance of the column of gel. Indeed, by using two adsorption chambers, the second containing twice as much gel as the first, it would be possible to recover 60% of the total hydrocarbon in the gas (or 76-77% of the total benzene in the gas) as practically pure benzene.

The coal gas used in the above experiment contained 0·80% of benzene and 0·19% of toluene (including xylene and naphthalene).

Experiments were next carried out to determine, more accurately than was possible in the small six-section column just described, the efficiency of adsorption and the composition of the recovered hydrocarbons when varying amounts of gas were passed over a constant weight of silica gel. The results of these experiments are summarised in Table VIII and are plotted in Fig. 7. The adsorption was carried out at a temperature of 14°, and the gas volumes passed are expressed as litres at N.T.P. per 100 grams of gel.

The first determination recorded, 273 litres of gas per 100 grams of gel, is for an adsorption which

was stopped exactly at the break point to benzene. For smaller quantities of gas it has been shown that all the benzene and all the other higher boiling hydrocarbons are quantitatively recovered. The important point therefore to be observed, where complete recovery of useful hydrocarbons is intended, is this break point to benzene. The still lower boiling constituents in coal gas, of which carbon bisulphide is the chief, are present at such small partial pressures and corresponding pressures (see Table V), that the amounts adsorbed are practically negligible. There is, therefore, in the hydrocarbon mixture recovered by silica gel none of the " pre-benzol " which must be so carefully fractionated from the spirit recovered by oil-washing processes.

Recovery of hydrocarbons from coal gas by silica gel.
o——o Adsorption as % weight of gel (× 10).
o—·—·— Composition by vol. of recovered liquid.
x—x—x Efficiency of benzene adsorption.
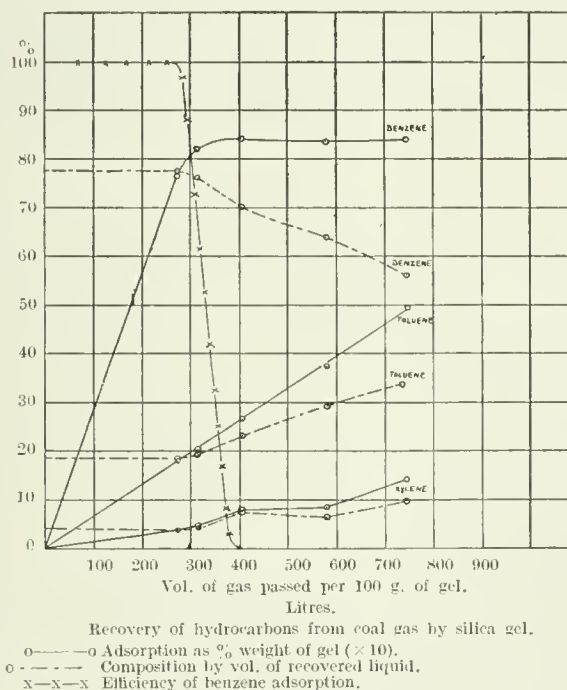
FIG. 7.

The recovery figures corresponding to a passage of 273 litres of gas represent, therefore a maximum recovery of the hydrocarbons valuable as motor spirit. Up to this point all such hydrocarbons are quantitatively adsorbed, and the proportions of the various hydrocarbons in the recovered mixture are therefore identical with the proportions in the original gas. If a greater volume of gas is passed over the

TABLE VIII.

Adsorption of hydrocarbons from coal gas by silica gel, showing the effect of ratio of gas volume to weight of gel on the degree of adsorption, distribution of individual hydrocarbons, and composition of the recovered benzol.

Expt. no.	Volume of gas per 100 g. of gel Litres N.T.P.	Total hydro-carbons adsorbed as \% weight of gel	Distribution of hydrocarbons as per cent. weight of gel Benzene	Toluene	Xylene	Composition by volume of liquid hydrocarbons recovered Benzene \%	Toluene \%	Xylene \%
1	273	9·84	7·65	1·80	0·39	77·5	18·5	4·0
2	315	10·70	8·20	2·04	0·46	76·2	19·45	4·35
3	405	11·89	8·41	2·70	0·78	70·2	22·4	7·7
4	578	12·95	8·35	3·75	0·85	64·0	29·4	6·6
5	743	14·80	8·40	4·96	1·44	56·2	33·9	9·9

same amount of gel, benzene begins to escape at the exit of the adsorber. The higher-boiling hydrocarbons do not, however, reach their break points for some considerable period later, with the result that the composition of the mixture of hydrocarbons recovered from the gel gradually changes, the percentage of benzene becoming smaller and that of the constituents of higher boiling point less. This is shown clearly by the broken curves of Fig. 7. From this series of curves it is possible to determine what proportion of gel to gas must be employed in order to secure any desired composition of hydrocarbons from the adsorption units. In order to secure complete "stripping" it is not possible to pass more gas of the composition under consideration than 273 litres per 100 grams of gel, and at this point the adsorption expressed as per cent. weight of the gel is 7·65% of benzene, 1·80 of toluene, and 0·39% of xylene (including higher-boiling hydrocarbons). The total effective adsorption of hydrocarbons is, therefore, approximately 10% of the weight of the gel.

It will be observed, also, that over the range of these experiments, which more than covers the limits within which a commercial adsorption process would be operated, no evidence was found of actual displacement of benzene by water vapour. The benzene having attained its maximum saturation value of 8·4% remained at this value over the whole period considered.

It is therefore practicable to use silica gel for the adsorption of hydrocarbon vapours from coal gas saturated with water vapour. The adsorption capacity for the hydrocarbon is, however, lower than if no water vapour were present, and in the above experiments amounted to slightly less than 10% of the weight of the gel.

The whole of the water vapour must be removed concurrently with the hydrocarbon vapour. This is unavoidable since water vapour is, as has been shown above, preferentially adsorbed.

The actual saturation value for benzene when adsorbed from coal gas is slightly higher (8·4%) than that obtained from the pure mixture of benzene, water vapour, and air investigated in the laboratory and illustrated in Fig. 6. This is to be expected since the benzene in the gel is dissolved in a certain amount of higher boiling hydrocarbons, which tend to lower its vapour pressure.

A very interesting observation from the point of view of motor fuel production was that the mixture of hydrocarbons recovered from the gel by direct distillation was of considerably better quality as motor fuel than the corresponding mixture obtained by scrubbing the gas with wash oil. Apart from the fact that wash oil takes up, under normal working conditions, only some 3 to 4% of its volume of hydrocarbons from the gas, the product obtained on distilling benzolised wash oil with steam (known technically as "light oil") contains, at the low-boiling end of the scale, appreciable amounts of carbon bisulphide, and at the high-boiling end some 5 to 10% of creosote or high-boiling paraffin oils distilled from the wash oil itself. The product obtained from silica gel contains a negligible amount

of carbon bisulphide, and clearly can derive no heavy oil from the distillation of the adsorbent. Furthermore, the light oil obtained by oil-washing is almost invariably yellow in colour, and quickly darkens to a light brown colour. The product obtained from silica gel, in the experiments described, was colourless, and remained colourless over several months. This is an important point for the motor spirit producer. It is not proposed in this paper to go into the causes of discoloration of motor spirit, but it is well known that there is frequently a close parallelism between discoloration of the spirit on storage and gum formation in the engine valves when the spirit is used. Drastic chemical treatment of the motor spirit, usually consisting in washing with concentrated sulphuric acid, is normally employed at the present time to remove these objectionable gum-forming constituents. Unfortunately such treatment destroys more than the objectionable constituents which it is intended to remove, and probably one-half to one million gallons of potentially valuable motor fuel are destroyed per annum in this country by motor benzol producers alone. Petroleum oil refiners are, on the whole, more enlightened in this respect, and by the development of less drastic refining processes have considerably reduced their refining losses. Nevertheless, the loss of valuable petroleum motor spirit by sulphuric acid washing probably greatly exceeds the figure given above for benzol.

The fact that the benzol recovered by means of silica gel appeared to show no signs of discoloration or resinification on storage was therefore an additional merit in the process, as it held out hopes that the product would need no further refining treatment. The other outstanding objectionable constituent in a motor spirit is sulphur, whether free or combined. In this respect the benzol recovered as described above did not show any marked superiority over the unrefined benzol as normally produced by oil-washing, except in so far as it was traceable to its freedom from carbon bisulphide. The average sulphur content of the gel recovered spirit was 0·4—0·5%. This statement is not in opposition to the claim of the Silica Gel Corporation that the sulphur content of many petroleum oils can be reduced to a very low figure indeed by treatment in the liquid phase with silica gel. The question of what types of sulphur compounds are removable by silica gel is a complex one, and it is not proposed to deal with it in the present paper.

The results already indicated warranted a trial of silica gel as an adsorbent for motor benzol production on a larger scale. The main points in favour of such an adsorbent in place of wash oil appeared to be :

(1) Adsorption of 10% by weight or about 11·5% by volume of hydrocarbons as compared with 3–4% by volume usually obtained with wash oil.

(2) Complete recovery of the useful hydrocarbons, very rarely obtained with wash oils.

(3) A crude oil free from low-boiling "pre-benzol" and uncontaminated with high-boiling creosote oils, except for the small amount adsorbed from the gas.

(4) The possibility of eliminating all chemical refining processes and particularly the sulphuric acid treatment.

(5) A saving, consequent on (4), of some 5–10% of the crude spirit.

(6) Avoidance of the nuisance and waste arising from the acid sludge produced by acid refining.

(7) A complete removal of naphthalene from coal gas is incidental to benzol recovery. This is of considerable importance to manufacturers of coal gas for town distribution. If desired a very small silica gel plant could be arranged to remove all naphthalene without stripping the gas of benzol.

It was at this stage that we were approached by Mr. J. A. Reavell, of the Kestner Evaporating and Engineering Company, the concessionaires of the Silica Gel Corporation, Baltimore, who informed us that they were about to carry out semi-large scale trials on the recovery of motor spirit from coke-oven

of vapours a bed type absorber, consisting of static beds of granular gel, was originally proposed and is still recommended for certain purposes. Where large volumes of gas are to be handled, however, a continuous circulation plant is used. In this case the gel is finely pulverised to about 200-mesh, and as circulated with the gas, which, by means of a pump, as maintained at a velocity sufficient to carry the gel along with it.

Adsorption is carried out in vertical adsorbers consisting of nests of tubes in an outer cooling jacket similar in construction to a tubular condenser. The gas and gel are separated at the top of each adsorber by a cyclone separator. The gel is fed into the gas stream at the bottom of the adsorber by means of a small screw conveyor. In actual practice three such adsorbers are used in series, the gas and gel flowing counter current through the series. In this way most effective " stripping " of the gas and saturation of the gel are secured. After traversing
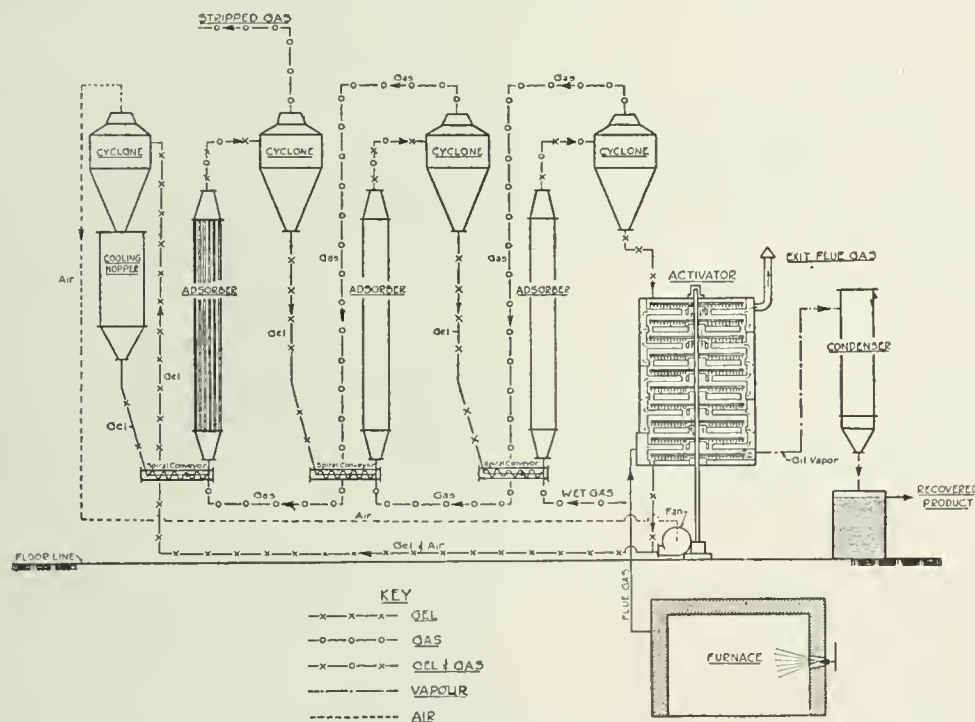
Reproduced by courtesy of the Silica Gel Corporation.

Fig. 8.

gases by silica gel adsorption. The National Benzole Association was invited to send representatives to investigate these trials and Mr. Basil Sadler and the author were appointed for that purpose. Together with Mr. Reavell they visited America during June and July last, and the remaining portion of this paper is devoted to a description of the trials carried out during that period.

The Silica Gel Corporation had during the past four years been experimenting on the industrial application of silica gel to the recovery of vapours and for treatment of liquids. For the adsorption

the final adsorber (the one nearest to the gas inlet), the saturated gel is taken from the cyclone separator by a screw conveyor to the top of the " primary activator." This is in effect a still in which the adsorbed vapours are driven off. It consists of a series of horizontal hearths placed vertically above one another and heated by the flue gases from a gas furnace. The hot gases pass through flues interspaced with the hearths but do not come in direct contact with the gel or distilled vapours. The gel entering at the top hearth is raked slowly downwards from hearth to hearth by means of revolving

rakes keyed to a vertical central shaft. The arrangement is very similar to the usual pyrites burner. The gel, completely freed from volatile vapours, is discharged from the bottom hearth into another screw conveyor which carried it to a " secondary activator." This activator consists of a slightly inclined revolving drum heated by means of an external jacket, through which hot flue gases pass. The purpose of this second activator, into which a small volume of air is drawn, is to burn off any non-volatile deposits from the gel. The temperature in the primary activator is maintained at 300°—350° C. and in the secondary activator at 550°—600°. The gel leaving the secondary activator is completely revivified and is returned by means of an air blast, provided by a fan working in a closed cycle, to the

80—100 cubic feet of gas per minute. This gas was drawn from the mains of the Bethlehem Steel Corporation's coke ovens at Baltimore at a point immediately following the ammonia scrubbers. The coke-oven plant was of the Koppers type and the gas had passed through the usual tar extractors, ammonia saturators, and water-spray coolers ; it may therefore be taken as a fair average coke-oven gas.

The amount of recoverable hydrocarbons in the gas was determined by passing a metered volume of the gas through a calcium chloride drying tube and then through a silica gel adsorption tube containing 3 lb. of gel. The adsorbed hydrocarbons were distilled off with steam at 200° C. and measured. This test was shown to be more rigorous than a determination made by the usual method of bubbling

Fig. 9.

gel storage and cooling hopper from which it again enters the adsorption cycle. The coke-oven gas, which passes through the adsorption unites in the opposite direction to the gel, is finally discharged from the last cyclone (the first from the point of view of gel flow) and passes through bag filters which remove any gel remaining in suspension and return it to the system.

The general lay out of the plant is shown in Fig. 8, which is reproduced by the courtesy of the Silica Gel Corporation. This does not show the horizontal secondary activator, the arrangement of which is shown in Fig. 9.

The adsorption plant erected at the time of our visit was an experimental plant, designed to handle

the gas through ice-cold wash oil. The content of hydrocarbons in the gas issuing from the adsorption plant was determined in the same manner, and both inlet and outlet tests were made concurrently with a quantitative examination of the hydrocarbons actually recovered in the plant. The results of a typical test are shown below :—

1. Benzol in inlet gas (control)
   Total volume passed .. .. = 54·53 c. ft. at 90° F.
   Total benzol recovered on distillation .. .. .. = 51·5 c.c.
   Benzol content (at 90° F.) .. $= \dfrac{51 \cdot 5 \times 0 \cdot 2200}{5 \cdot 463}$ Imp. gals. per 10,000 c. ft.
   = 2·073 Imp. gals. per 10,000 c. ft.

II. Benzol in effluent gas from plant (control).
Volume gas passed ... .. = 186·68 c. ft. at 90° F.
Benzol recovered .. .. = 7·5 c.c.

Benzol content (at 90° F.) .. = $\dfrac{7 \cdot 5 \times 0 \cdot 2200}{18 \cdot 668}$ Imp. gals. per 10,000 c. ft.
= 0·0884 gal. per 10,000 c. ft.

III. Efficiency of adsorption in plant (calculated from control tests).

$$= \frac{(2 \cdot 073 - 0 \cdot 0884) \times 100}{2 \cdot 073} = \frac{1 \cdot 9846}{2 \cdot 073} = 95 \cdot 7\%$$

IV. Recovery of crude benzol in plant.
Over the same period as that of the above tests.
Gas treated .. .. = 103,505 c. ft. (at 90° F.)
Recovered crude benzol .. = 10·55 Imp. gals.
Benzol .. .. .. = 1·898 Imp. gals. per 10,000 c. ft.
Theoretical recoverable .. = 1·9856 gals. per 10,000 c. ft.
Efficiency of recovery.. .. = 95·6 per cent.

This figure of 95·6% is the average of three shifts continuous working ; actually one of the shifts operated at 97% efficiency.

The hydrocarbons recovered from the effluent gas had an unpleasant odour, and boiled at 76°—78° C. A detailed examination of this product was not made, as the quantity available was too small ; it is almost certain, however, from its general properties, that only a small portion of this fraction could possibly be considered useful as motor spirit. Estimating this proportion at a liberal figure of 50%, then the total loss of motor spirit in the effluent is only 0·044 gallon per 10,000 cu. ft., and the efficiency of adsorption of motor spirit in the plant becomes 97·9% of the theoretical.

There is therefore an adsorption of motor spirit from the gas of 97·9% efficiency, followed by a recovery of liquid benzol from the gel of 95·6% efficiency, giving a total all-round efficiency of recovery in the plant equal to 93·6—95% of the amount initially present in the gas.

These results entirely bear out the conclusions arrived at in the laboratory as to the excellent recovery of hydrocarbons attainable. The temperature conditions of the test were, moreover, not favourable, the temperature of the inlet gas being 90° F. and that of the cooling water 80° F.

The recovered product was, however, not so satisfactory. A colourless, non-gumming distillate had been expected from the gel, but the product actually obtained was yellow or yellowish-brown, and neither modifications in the method of working nor redistillation of the crude spirit gave appreciable improvement. It was evident that the crude spirit would need further refining. The refining of a liquid spirit with gel had already been worked out in detail by the Silica Gel Corporation in connexion with the refining of petroleum oils and a continuous agitation and filtration plant capable of dealing with 3000 gals. per day had been designed to operate this process. It had been found that a strongly coloured gasoline, kerosene or lubricating oil when agitated with successive quantities of gel and filtered was rendered of excellent colour and practically free from sulphur. The case of benzol, however, proved to be somewhat different. Agita-

tion with silica gel followed by filtration did not produce marked improvement in the appearance of the product ; but there was this actual improvement in fact, that, if the liquid so treated were distilled, 88—89% came over below 130° C. as a colourless product, while the residual high-boiling 10% was only very slightly coloured. The higher-boiling fraction could also be rendered colourless by a further treatment with gel, and redistillation.

This refining action of the silica gel in the liquid phase appears to be due to the rapid polymerisation of those constituents in the crude benzol which are the cause of discoloration. It is already known that if a crude benzol is allowed to stand for a long period these constituents tend to polymerise spontaneously, and that if such a "matured" benzol is distilled, the high-boiling polymers remain as a gum-like residue in the distillation vessel. Silica gel by adsorbing such constituents increases their activity and accentuates the process of polymerisation, with the result that after a very short period of time the resin-like products can be completely separated by distillation.

The sulphur content of the spirit is not greatly reduced by this liquid phase refining process. The actual content of sulphur was 0·37% in the crude, and 0·28% in the refined spirit. There is therefore greater difficulty in removing the sulphur, whether free or combined, from benzol than from petroleum, for which the Silica Gel Corporation reports almost complete removal of sulphur by simple agitation with gel.

The plant designed by the American Corporation for carrying out the liquid phase refining treatment described consists in its simplest form of a tank fitted with an agitator for mixing the liquid and gel. The mixture from this tank runs to an Oliver continuous vacuum filter from which the treated liquid is withdrawn, while the gel is carried by screw conveyor to a primary and secondary activator of the type already described. In practice, in order to obtain the maximum duty from the gel, three agitating vessels and three Oliver filters are placed in series, the gel and liquid passing countercurrent through the system.

The cycle of storage tanks, agitating tanks, Oliver filters and activators is made gas tight so that losses by evaporation are reduced to a minimum. The actual loss of spirit during this process is confined to the very small amount of the resin-forming constituents which are polymerised, the vapour which escapes through the vacuum pump operating the filters, and the vapour which escapes condensation after being distilled off in the activators. These losses amount to 2% as a maximum and probably less than 1% of the spirit treated. They are to be compared with the much greater loss which occurs when a sulphuric acid refining treatment is employed. The general arrangement of a continuous liquid refining plant is shown in Fig. 9.

The crude spirit after agitation with gel and filtration is distilled. This distillation is a necessary stage in the refining process, but it also serves for

the fractionation of the product into motor spirit and solvent. As the quantities handled in our trials were small, two fractions only were obtained, viz., at 80—130 C. and at 130°—184 C. Table IX. shows the efficiency of the recovery, refining and distillation operations, and the over-all efficiency of recovery based upon the amount of hydrocarbons initially present in the gas. It will be seen that of 100 parts of hydrocarbons in the gas 87·8 are recovered, of which 80·8 are obtained as refined motor spirit and 7 parts as solvent.

TABLE IX.

Stage	Recovery % during stage		Recovery % of initial amount in gas
	%		%
(1) Adsorption	97·0	..	97·0
(2) Distillation of gel	95·6	..	92·7
(3) Liquid-phase refining ..	98·8	..	91·0
(4) Fractionation—			
(a) Motor benzol (80°–130° C.)	88·8	..	80·8
(b) Solvent (130°–184° C.)	7·7	..	7·0
(c) Residue and losses ..	3·5	..	—
			87·8

The quality of the motor spirit recovered was excellent and able to meet the requirements of the " British Engineering Standards " specification No. 135, 1921, except in one important particular. The specification requires that a satisfactory motor spirit shall give only a pale yellow coloration when treated with concentrated sulphuric acid. This requirement is, in fact, a test that all the unsaturated constituents of the benzol have been eliminated. Silica gel does not remove all the unsaturated constituents, but only those which are objectionable because of their tendency to polymerise. This discrimination between valuable and objectionable unsaturated compounds is one of the chief merits of the process, and provided that the product obtained is able to prove its suitability for use in a motor engine, the fact that it is unable to pass the sulphuric acid test is of no consequence, and should be ignored. Alternative laboratory tests are available which more truly check the satisfactory quality of a motor fuel. The ultimate test must be satisfactory performance in a motor engine, and in such a test made upon the gel-recovered and refined spirit obtained as described above, the result was fully equal to that obtained on a first-quality acid-washed product. In one respect the silica gel product is likely to prove superior to the acid-washed product. During the acid refining process compounds are frequently formed of unsaturated hydrocarbons with the sulphuric acid, which appear to be neutral sulphuric acid esters, soluble in the benzol and not removable by either water or alkali washing. At high temperatures these decompose with liberation of sulphur dioxide, which causes corrosion of the metal parts with which it comes in contact. A product refined by silica gel contains none of these compounds, and is therefore free from one of the objections which may apply to a spirit refined with sulphuric acid.

Table X. summarises the results of a 44-hour trial run made upon two similar Studebaker Light-6 1922 engines, installed in the laboratory and operating through flexible couplings, two compound-wound generators, the loads upon which were controlled by means of adjustable plates in a water-resistance box. One engine was operated on the motor benzol recovered and refined by silica gel, the other on a good quality acid-refined spirit. Both engines were taken down and thoroughly cleaned before the test.

TABLE X.

Comparative engine tests between Bethlehem acid-washed benzol and " silica gel " benzol.

	Bethlehem		Silica gel.
Time, hours	44·5	..	44·5
Fuel consumption :—			
Gallons	67·25	..	65·75
Lb.	490	..	480
Air : fuel ratio, lb. per lb.	10·2	..	10·4
R.p.m.	1000	..	1000
Load, h.p.	13·1	..	13·1
Intake water, °F.	120	..	117
Difference, °F.	50	..	49
Crank case, °F.	166	..	165
Lubricating oil, qts. ..	2·1	..	2·3
Carbon on exhaust valves, grams	0·0800	..	0·0402
Carbon on intake valves, grams ..	0·8600	..	0·5903
Carbon on cylinders and pistons, g.	25·5189	..	28·6714

The gum formation was extremely low in both engines. No gum was found in the valve parts, and the small amount shown was found on the six intake valve heads and stems. Both products showed very little gumming indeed, and what advantage there was appeared to lie with the silica gel product ; both sets of valves were, however, in very good condition. The carbon deposit in the exhaust valves was negligible in both cases.

The amount of carbon deposited in the cylinders was approximately the same in the two cases, with a slight advantage (25·5—28·7 g.) in favour of the Bethlehem benzol. This difference, however, was not more than might be obtained with runs on identical fuel in the same engine, and, in any case, this deposit is probably largely due to the lubricating oil rather than the benzol. On the whole, this comparative test showed very little difference between the acid-refined and silica gel products, with, perhaps, a slight advantage in favour of the silica gel with respect to gumming in the intake valves.

Both engines ran smoothly throughout the whole period, and no trouble was experienced after occasional stoppages. The quality of the products produced has therefore been shown, as far as can be shown on such a scale, to be entirely satisfactory as a motor fuel. The yield from gas is also superior to that obtained by oil-washing, while the refining losses are considerably reduced.

The ultimate test of the commercial value of the process lies in the cost of production. It is difficult to come to a satisfactory conclusion on this point until trials on a larger scale have been carried out, but all the calculations that we are in a position to make show that a very real reduction in cost of production is to be expected. The plant, although mechanically somewhat complicated in comparison

with an oil absorption or static bed type apparatus, requires extremely little supervision and, once started, is practically automatic. The circulation of the solid gel, by means of conveyors and air blasts, works smoothly, and no sign was shown of any appreciable wear in the moving parts. No trouble was experienced during the trials made due to the presence of impurities such as tar fog, which, it had been anticipated, might coat the surface of the gel and so inhibit adsorption. The adsorption of water vapour by silica gel, though undesirable from the standpoint of hydrocarbon recovery, does not prevent adsorption of hydrocarbons, though the degree of adsorption is limited to 9—10%, and in practice is calculated not to exceed 8% of the weight of the gel.
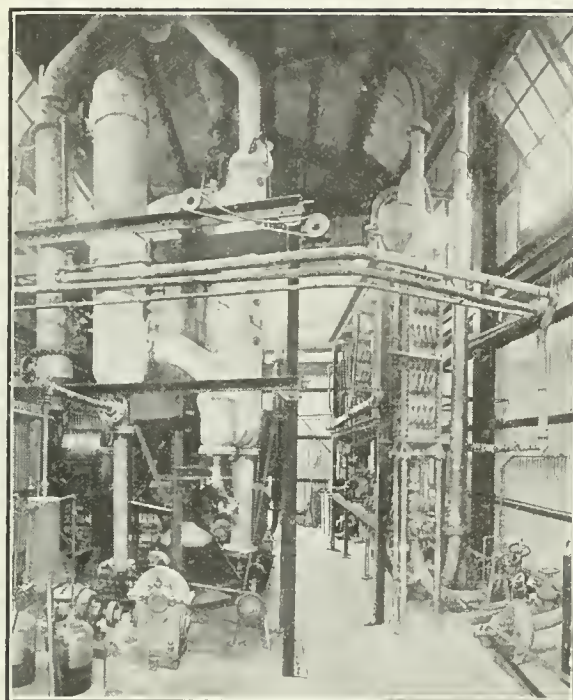

Fig. 11.

fore the vertical activator; it will then pass through the three small units of the liquid refining plant, and could, if required, return then to the vertical activator of the adsorption cycle. Actually it is more convenient, for various reasons, to provide a small independent primary activator for the gel which has been by-passed for refining purposes, after which the gel again joins the main stream through a common horizontal activator. This general arrangement is shown in Fig. 10, which is reproduced by courtesy of
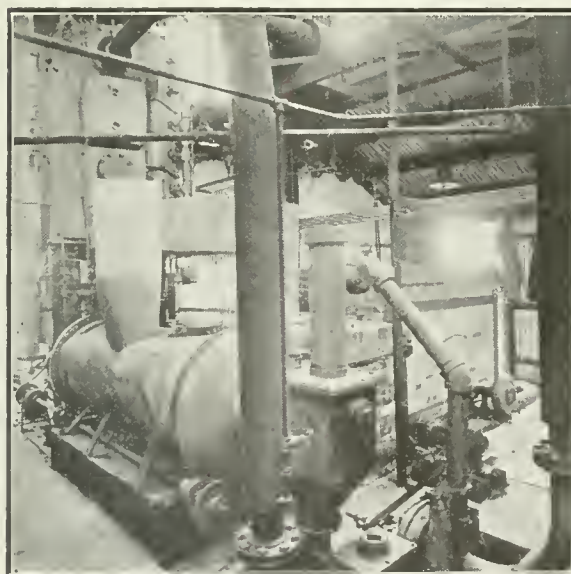

Fig. 10.

The process offers such advantages that it appeared justifiable to adopt it on the commercial scale for benzol recovery at a coke-oven plant, and arrangements are being made in this country for the installation of a full-scale plant for this purpose. The results of this trial will be awaited with the greatest interest. The plant will consist of combined adsorption and refining units. The amount of gel required for refining is a mere fraction of that required to recover the benzol from the gas (about 3%) and, as arranged, the refining section will be a small addition to the recovery units. The gel required will be drawn from the adsorption cycle at a point immediately be-

Fig. 12.

the Silica Gel Corporation, and represents the pro-
posed plant for the first industrial-scale recovery
of refined motor spirit from coke-oven gas by the
silica gel process.

The author has confined his attention to one indus-
trial use of silica gel. It appeared preferable to
discuss one application in detail rather than generalise
over a large range of possible applications. It is,
however, evident that silica gel can be applied to a
very large number of industrial problems, whether
as an adsorbent for vapours, as an adsorbent for
substances in solution, as an accelerator of chemical
reactions, or as an inert carrier for other catalytic
substances. There is a danger of overstating its
capabilities ; silica gel is not a philosopher's stone—
it is simply a resistant material possessing enormous
surface and fine pores.   There is a great industrial
field in which such properties will be of value, but
progress can only be made by careful scientific
examination of each problem as it arises.

APPENDIX.

The three photographs (reproduced by courtesy of
the Silica Gel Corporation) show the semi-large scale
plant upon which the trials described in the text
were carried out :—

Fig. 10. Cooling hopper for gel and adsorbers.

Fig. 11. Gas meters and adsorbers on right ; con-
densers, separators, and bottle receivers
on the left.

Fig. 12. Horizontal secondary activator, discharg-
ing in the foreground into the gel circu-
lation system.   The vertical primary
activator can be seen above and beyond
the secondary activator.

DISCUSSION.

MR. GORDON ADAM (Gas Light and Coke Co.),
who opened the discussion, said there was no doubt
room for both the processes dealt with in the two
papers. Most benzol manufacturers would agree
that benzol extraction and refining was not nearly so
efficient as it might be. The existing process for
the extraction of benzol was the oil washing process,
and there was also the compression process which
was used mostly in the United States, but the
efficiency of the existing oil washing process, used in
this country, would be about 85 per cent. That
would represent a good up-to-date plant, but the
steam consumption was high. About ten times the
weight of steam was required for the distillation of
the benzol out of the wash oil compared with
something like four times the weight in the case of
the charcoal process. It was not possible to put
the steam consumption in the case of the silica gel
process into actual figures. In any case, it would
have to be in pounds of coal per thousand gallons
of spirit and perhaps later on some figure might be
given for this. The silica gel process certainly
offered a big improvement over the existing method
of refining benzol. From the point of view of the
chemical engineering of the problem, the two
processes which had been described represented

the static and the dynamic processes. The silica
gel, from its nature, could be handled in a different
way from charcoal. Being practically a sand it
could be put through various complicated mechanical
processes without losing its efficiency, whereas the
charcoal, being practically carbon must be used in
the static condition. That was an advantage to
the silica gel process. Possibly for small plants the
static gel method would be most adopted, but there
came a point where the charcoal process would be
cheaper in capital cost than the static gel process,
whereas for very large plants this dynamic process
would probably be found to be cheaper in first cost.
Looking at the charcoal process from a gas company's
point of view, Major Gloag had mentioned a new
process for the removal of sulphuretted hydrogen.
That seemed to be a most interesting application
and he looked forward to further information on
that at a later period, especially in the application
of the process to coke oven plant. He would like to
know how much ammonia was required for the
catalytic reaction in the conversion of H_2S to sulphur.
The blowing in of ammonia, as mentioned by the
author, seemed to him to render this rather an
expensive process. He knew that gas works people
usually slipped in a little ammonia to activate the
oxide in the purifiers, but the amount so used was
about five grains per thousand cubic feet of gas. If
a larger quantity was required, then it would make
the process expensive. The other application of
the process to gasworks processes appeared to be
in the removal of the naphthalene and also the
carbon bisulphide. There was an existing process
for the removal of carbon bisulphide which he
believed was rather expensive but it would appear
that the activated charcoal would certainly be a very
cheap method for the removal of carbon bisulphide
and for reducing the sulphur content of the gas.
Both processes could be applied to the removal of
sulphur, and the silica gel process could also be
applied, possibly, to the removal of water vapour
from gas. He understood that gas engineers had
recently become very much alarmed about the
corrosion of their pipes. They had found that after
the pipes had been down a certain number of years,
serious corrosion occurred and it had been suggested
that the removal of the water vapour from the gas
might lessen this corrosion of the pipes. The silica
gel process appeared to be a cheap means for this
purpose. There was really room for both the
processes, and the question came down finally to
what is the difference in cost of a gallon of benzol
produced by the silica gel process and by the charcoal
process as against the existing processes. Perhaps
there might be some enlightment on that point.
Personally he could not imagine that the existing
oil washing plants would be scrapped in favour of
either the silica gel or charcoal processes, but where
new plant was being put down then there might
well be keen competition on the part of either silica
gel or charcoal.

DR. W. R. ORMANDY said it was a matter of great
regret to him that the paper dealing with activated
carbon was not on more scientific lines. It was
true that as a technological society they were anxious

to know what had been done on the large scale, and the mere fact that the large plants which had been referred to had been put in and were running, was in itself very strong and very cogent evidence that the process is a workable one and also commercial, but there was this to bear in mind, that in Germany the economic conditions—the political economic conditions—were vastly different from those obtaining in the rest of the world, and in quite a number of cases processes which might prove to be financially exceedingly good in Germany would be much less economical in the rest of the world for the reason that the Germans were able to produce internally the necessary materials, and they were in consequence using heavy fuels for some of these processes which in this country could not be sold because they would be compared to the lower prices for more easily obtainable and more easily applicable fuels. Moreover, in Germany there was a fairly heavy duty on motor spirit going into the country, and therefore the Germans had a very great advantage from the economic standpoint. For these reasons he hoped Major Gloag would be able, at some future date, to give, as an adjunct to the present valuable technical paper, one on more theoretical lines dealing with the fundamental scientific considerations underlying the principle employed in the method which had been described. He had been particularly interested in the small experimental plant referred to by Major Gloag for the production of benzene and adsorbable gases and liquids, because the internal cooling was carried out with very strongly non-conducting material. The author said that the distance between the coils was something like 10 cm. He personally had had a similar problem in applying heat to bodies adsorbed in kieselguhr, and his experience had been that a distance of 10 cm. was hopelessly big and that a distance of 2 cm. was the one which gave the best results. The question of back pressure also arose in connection with the use of comparatively heavy beds of charcoal, and he would like to know what back pressure was obtained in the case of a 2 metre bed of charcoal where gas was being passed in at the top and came out at the bottom. The question of fire had never arisen, but it was well known that wherever benzole passed through a pipe and there was a liquid friction against the metal, it was essential that there should be no broken pipe, and that the benzol should not run from one vessel to another without the vessels being electrically connected. The process of removing sulphur was of tremendous interest, and it would be of much greater interest in the future than it had been in the past because there was not the slightest question that when the technique of high pressure catalytic reactions was fully developed, very many catalytic reactions would be carried out under high pressures using as raw materials CO, CO_2, hydrogen, water gas, producer gas and similar bodies in which the sulphur was always the one vital difficulty. It was always the sulphur which acted as the destroyer of catalytic surfaces, and a dynamic process was required which would remove the sulphur down to minute quantities at a reasonable price. In the processes in which hydrogen

was the factor, the cost of pure hydrogen was usually the deciding factor in the economic value of the process, and the cost of hydrogen was largely dependent on the price paid for the removal of the sulphur. In the case of the recent process of the Baeyer Co. for the production of synthetic methyl alcohol, which involved the use of water gas plus hydrogen under very high pressures and fairly high temperatures, one essential factor was the removal of the sulphur, but there were always traces of oxygen and also ammonia in the gas, and he would like to know the composition of the benzol recovered from the activated carbon process. Did the benzol so produced, after it had been fractionated, contain elementary sulphur. With regard to the presence of sulphur in motor spirit, 0·4 per cent. was the allowance per gallon, but the important point was the presence of elementary sulphur and an exceedingly small trace of that was of vital importance. Sulphur as carbon bisulphide was of no importance in that it did not corrode the engine or valves. It only corroded anywhere where the temperature was below 100° and condensed water was present. One of the tests of a motor fuel was that on evaporation it should not leave more than a slight discolouration of copper, and to get that result it was essential that there should not be more than 0·1 milligramme of elementary sulphur per 100 c.c. of liquid. With one milligramme, the copper would go jet black, and with two milligrammes it would peel and copper sulphate would be formed. Therefore, it was the elementary sulphur which caused the trouble. Whether the activated carbon acted merely by virtue of capillaries from the physico-chemical standpoint, there was no doubt whatever that although it had exceedingly minute pores it did not act merely as silica gel was said possibly to act. It was undoubtedly selective in its operation. He knew of one case in which nine de-colourising carbons were used in succession for removing the colour from a certain aqueous solution without the slightest effect, but one of the special Baeyer carbons in small quantities gave a clear water white solution, which showed that the activated carbon was selective in its action.

In the case of both charcoal and silica gel the unsaturated state of the body being adsorbed played a part. Unsaturated bodies would be more readily adsorbed than saturated bodies. That, perhaps, was a theoretical point which could not be discussed on that occasion. He would also like to ask Prof. Williams the same question that he had asked Major Gloag, viz., was there any elementary sulphur in the final product obtained with the silica gel process, as distinct from combined sulphur which, as he had already stated, he was not particularly concerned about. He would like to know how long the silica gel lasted. If the gel was going to be heated up to 620°, which was just dull red heat, to burn out the polymerised hydrocarbons which had settled in the pores, he would have thought that in any reasonable period of time, in the presence of a small amount of air, it would be a mighty difficult thing to burn out the hydrocarbons in the pores of the gels. There seemed almost the possibility that activated carbon would be produced.

Major GLOAG, replying to the comments made so far, said that the ammonia which was fed in, mentioned by Mr. Adam, was fed in with a certain degree of accuracy, in proportion to the amount of H₂S in the gas. The ordinary Ruhr coal gas contained about 35 grammes of H₂S. per cubic metre, and 1 per cent. or 0·5 gramme of ammonia per cubic metre gave a perfect deposition of the sulphur in the pores of the carbon. The steam consumption had been brought down to 4 lb. of steam per lb. of benzol distilled, but, working on 100 per cent. recovery, it would come down to 3 or 3½ lb. He agreed that owners of oil-washing plants in this country could not be expected to scrap them at once, especially if they were working with a reasonable efficiency. It was rather difficult to reply to Dr. Ormandy. Such a lot of papers had been written on activated carbon by various people in Germany and America that he did not want to repeat all the questions that had previously been dealt with in the technical papers. He therefore thought it would be to the best advantage of the members of the Group to bring forward an account of what is actually being done in the practical application of the process. He would have to leave it to others more qualified than himself to deal with the theoretical side. At the same time, probably in two or three months' time, some very fine papers were likely to be published on the theoretical side of adsorption by activated carbon.

Dr. ORMANDY said the point was that the various articles that had been published on this aspect differed enormously, and what would be of advantage would be scientific data relating to these particular carbons referred to by Major Gloag, and not general papers.

Major GLOAG said that perhaps that would be dealt with at a later date. With regard to the distance apart of the coils, he was interested to hear what Dr. Ormandy had said. There might be special conditions which affected the distance apart of the coils. At a distance of 10 c.m. there was quite a good heat exchange with the grade of coal used in this instance, but he could not say what it would be with other fuels. It might be necessary to alter the distance. With regard to back pressure, Mr. Hoffard, of the National Benzole Association, had worked out some figures relating to a metre depth of coal, and there appeared to be about ¾ inch water pressure loss with a speed of about 2000 ft. per hour. With a speed of about 16,000 ft. per hour, the loss was something like 7 in. through a depth of 1 metre of coal. There was elementary sulphur present with gas works practice and gasworks purifier, but with active charcoal purification taking H₂S out, it had yet to be proved whether there is elementary sulphur present. He hoped to have figures soon as samples had been tested by the Gas Light and Coke Co.

Prof. WILLIAMS, also replying to the discussion up to this point, agreed that the question of the steam consumption with the silica gel process had better be left in abeyance for the moment because the figures to be obtained from the small plant at his disposal would not be of any great value. As to the theoretical side of the silica gel process, he had not put forward the purely capillary theory because he believed in it. As a matter of fact, he thought there were very big holes to pick in it, but it gave a more or less concrete picture and something to work on for the purposes of a technical discussion. He agreed with Dr. Ormandy that a purely capillary theory would not solve the problem entirely. There was obviously selective adsorption, but the whole thing at present was so involved that it was impossible to say that any part of the action was capillary and any part was molecular. With regard to the removal of water from coal gas, silica gel would dry coal gas, and it had also been used as a dehydrating agent for drying blast furnace gas for getting a higher temperature in metallurgical operations, but he was not sure whether large plants had been put up for it. The Chairman had just mentioned to him, however, that plants had been specifically installed for drying blast furnace gas.

Dr. ORMANDY : In other words, it will be cheaper to dry coal gas first by silica gel.

Prof. WILLIAMS said that was what he was coming to. With regard to the working cost, which was the ultimate test of any process, he would like to have dealt with that, but he would not presume to put forward a complete working cost sheet of a plant which he had examined only to the extent of about 100 cub. ft. per day. It would not have been fair. The question of costs had been carefully gone into, however, and an endeavour had been made to compare the costs with those of the oil washing process. At the same time, those interested in benzol and by-product works in this country knew how extraordinarily difficult it was to get anything like a scientific costing system in relation to the production of by-products. There was no real standard basis of calculation. In circumstances like this it was exceedingly difficult to reach a cost basis, but a figure of cost had been arrived at which a coke oven manager had agreed was a fair one. That was 2·3d. per gallon, but it was an estimated figure, as the saving to be effected by the use of silica gel. Anyway, it was a figure to take hold of and remember for the purpose.

Dr. Ormandy had raised such a large number of points that it was clear there was subject matter for three or four papers. He entirely agreed that sulphur was the bugbear, and he also agreed that the real test of benzol for motor car purposes was the corrosion of copper. As to tests, Mr. Hoffard of the National Benzol Association was now working out a method which was to be standardised by the Association, for dealing with the sulphur.

Dr. ORMANDY : A method has been worked out and published for estimating the elementary sulphur.

Prof. WILLIAMS said the method which he had used was to boil 100 c.c. over a water bath for half-an-hour or an hour, and then examine the copper dish, but he did not put that forward as a sound basis for a real test for a motor-car spirit. The benzol obtained with silica gel had been subjected to the copper dish test to see if there was any corrosion and any deposit of gum on the copper surface.

Dr. ORMANDY said that in addition to corrosion there was also a black shiny varnish on the inside of the dish in the case of gummy fuels. He believed some dishes were present and could be inspected.

Prof. WILLIAMS, referring to the escape of toluol and xylol, mentioned by Dr. Ormandy, said that a reference to Fig. 7 would show the composition of the distillate. The amount of toluol and xylol adsorbed was also shown, and it was evident that there was no loss of these over the range normally worked. He did not think silica gel would be suitable for removing water in the sense that there would be any advantage gained. He had put forward a proposal of that kind, but the Benzole Association did not consider it a commercial proposition. As to the variation in colour of the product of the laboratory and that produced on the large scale, the reason suggested by Dr. Ormandy he believed was somewhere near the mark. It had been mentioned in the paper that when the gel had been subjected to coke oven or coal gas it was practically colourless. In this he was referring to his own experiments and not the experiments in America. When it was heated slowly the benzol went a brown colour, the temperature being continued to 110 or 120 deg. He believed it was due to the polymerisation of the very highly unsaturated hydrocarbons which were supposed to be the cause of the gumming. By their adsorption in the gel they were made more chemically active themselves and the process of polymerisation was accelerated by the fact of their being adsorbed, their chemical energy being increased in the adsorbed layer on the surface of the gel. The reason for not getting colourless products on the works scale was that sufficient time had not been given for this polymerisation to take place. In the laboratory there was slow heating and distillation by steam, and after the material had had time to polymerise there was distillation and the polymerised products were left behind. In the works distillation took place before there had been time for polymerisation to take place, and the things were then present in the liquid motor benzol. It was all a question of the difference between gentle heating and quick heating. The high boiling 7 per cent. solvent referred to was perfect in colour, and was not rejected on account of quality but because he wanted to get the yield of motor spirit of the range specified by the Benzole Association. That solvent was perfectly good stuff and was not wasted at all.

Mr. F. M. POTTER thought it was entirely wrong to regard the charcoal and the silica gel processes as in every case competitive. In the case of water removal, for instance, it seemed to him, although he had not closely studied it, that silica gel was pre-eminently the material to be considered. On the other hand, if we took another important series of questions which had not been considered in very great detail during the discussion, viz., the recovery of volatile solvents from air/solvent mixtures, the activated carbon process had a very high efficiency when applied to low concentration mixtures. The plants working in Germany on solvent mixtures seemed to work quite smoothly, and in order to

compare them with the silica gel process he would like to stress again the question of cost, which had already been mentioned. There was the question of the royalty to be charged by the holders of the silica gel patents and the charges to be made for the adsorbent material. We had been led to assume that the cost of silica gel would be cheaper than carbon because it was made from a relatively cheap raw material—water glass. Whether, when the whole process had been worked out, that would be found to be so, he did not know. The proof of that would be at what price can the user obtain this solid adsorbent and what royalty had he got to pay on the process? He did not know whether it would be possible, that evening, to obtain figures for that. In the case of the recovery of volatile solvents from air, he had some figures for various cases. For instance, in the case of alcohol/air mixture containing alcohol of the order of 30 grains per cubic metre, the steam used for recovery was 2 kg. per kg. of alcohol recovered. That was the steam used in the steaming process, the alcohol being recovered at 45 per cent. strength. It was then necessary to dry the charcoal, if the concentration was low, and to heat that to the temperature required—130°C.—which used up a further 4 kg. of steam. The alcohol then had to be rectified to 98 per cent. strength in order to bring it back into the process, and the figure used for this purpose appeared to be of the order of $1\frac{1}{2}$ kg. of steam per kg. of alcohol, so that the total recovery of 98 per cent. alcohol involved $7\frac{1}{2}$ kg. of steam per kg. of alcohol recovered. That was on a plant working with H charcoal, which was said to be not so efficient as the T charcoal. In the case of benzol recovery, there again it was necessary very definitely to separate the problem into the recovery of benzol from town gas, which had already passed through the oxide purifiers and was substantially free from H_2S and coke-oven gas. He had taken a sample of benzol from the Bernau plant, and it did not give any darkening whatsoever with the mercury test nor did it give any reaction with the copper dish. It gave a very slight trace of gum, of the order of 3 to 4 milligrammes, and was within the limits set by the American specification for that test. It showed no sign whatsoever of free sulphur. It contained carbon bisulphide, but he did not think the figures from any one batch at Bernau were reliable because it seemed to him that the figures of carbon bisulphide in the recovery of benzol depended entirely on the conditions under which the filters were operated. It was possible to work the charcoal filters under the same conditions as the gel process, in which the carbon bisulphide was passed forward and not recovered. He had been told, for what it was worth, that the carbon bisulphide in the benzol at Bernau was of the order of 0·4 per cent., and that it had been found possible to market the charcoal recovered benzol at 38 gold pfg. per litre as against 32 pfg. for petrol. Why that preference should be given he did not know, unless it was thought that the carbon bisulphide was an advantage. It was agreed that it was not a defect from the point of view of corrosion, and it was

possible that an advantage was found in an extra kick in starting up. The charcoal-recovered benzol from town's gas, on the basis of the distillation tests, certainly seemed to be very useful. On the other hand, the people in Germany did not seem to be very certain as to whether they should remove the sulphur. From some experiments which he himself had made, it seemed to be practically certain that it would have to be removed, because if there was sulphur in coke-oven gas it was certain that there would be sulphur in the recovered benzol. It was curious that there was a tendency for the benzol recovered under works conditions to be coloured, as compared with the water white liquid obtained in the laboratory, when using the silica gel process. On the other hand, charcoal recovered benzol was reasonably free from colour and under ordinary conditions would be quite marketable. With regard to gumminess, he very much doubted whether this really arose from the gas, and was inclined to think that we in this country had got a wrong impression of gumminess. In the ordinary way it arose, undoubtedly, in part from the deposition of the wash oil, possibly from the phenol content. Otherwise it was difficult to explain why the charcoal recovered benzol showed no loss of activity and did not seem to have any appreciable gum content.

Dr. LESSING asked whether it would be possible to use a silica gel—he knew carbon could be used in this way—with a slight covering of oil, and in that way to get the advantage of the action of the oil as a solvent added to the adsorptive quality of the porous silica gel. If the mechanical theory held, then this should be possible. Would Prof. Williams say whether he thought that at all possible ?

Mr. P. PARRISH said it must be perfectly evident to anyone who had listened to the papers and discussion that these two materials had their own specific functions to perform. He felt that silica gel had a very good function to perform in regard to the removal of naphthalene from coal gas, and if, as had been adumbrated, a small plant involving about 30 lbs. of silica gel could deal with about 2 million cubic feet of gas, and if the economics were right, then there was an enormous field for it. He would like to know if debenzolised gas was used as the medium by which the silica gel was blown through the tubes, and could Prof. Williams give any idea of the quantity of gas required in order to perform that particular function. Dr. Ormandy had referred to the deposition of carbon, and that was a very important problem in connection with any catalytic process or even in connection with any new process for the elimination of carbon bisulphide. The point raised by Major Gloag, with regard to the removal of hydrogen sulphide by means of activated carbon, was an important one, but he was afraid that he was not enamoured of the economic success of that process, because apparently the Baeyer Co. proposed to adopt a process which had failed from the economic point of view in France. He referred to the removal of sulphur by ammonium sulphide by the Gauthier and Ducancel process, which was worked successfully during the war. Large quantities of sulphur were

recovered in France by that process, but the whole of the large plant erected by the French Government had been scrapped, because it was not an economic proposition, and if Major Gloag could say anything with regard to the economics of the process proposed by the Baeyer Co., everyone would be interested.

Mr. SUTCLIFFE said he spoke as a manufacturer of carbon in this country, and he was astonished to find the varying results that could be obtained from different classes of carbon. One carbon would have wonderful de-colourising power and very low adsorption power, whilst another would have very high adsorption power and very low de-colourising power. He believed that silica gel could do certain things that carbon could not, but there were undoubtedly other cases in which carbon could do work which silica gel could not. Possibly a combination of the two would bring about very good results.

Mr. ERIC REAVELL asked Major Gloag what was the method of control of operation with regard to alcohol recovery—*i.e.*, how was it known when the filter was saturated with alcohol in order to avoid any loss through the filters ? Again, in the removal of naphthalene from the pre-filters, it was stated that the method · of burning the carbon out had been abandoned and that a solvent was used. Had Major Gloag any figures of the loss of solvent which would occur in removing the naphthalene and its impurities. Also, could Major Gloag give any information as to the influence of temperature on the gases entering the filters, because in the case of oil washing there was a loss of efficiency as the temperature of the gases increased. Again, was any information available as to the efficiency of recovery in the case of town gas that had been used on the Leverkusen plant, and had any experiments been carried out on coke-oven gas.

Major GLOAG, in his final reply to the discussion, said he would give Mr. Potter the figures he wanted as regards cost in his office, but he did not want to broadcast them. As regards the removal of CS_2, that was adsorbed by activated carbon and completely recovered. In the actual case of benzol recovery they worked slightly above the break point, so that all the CS_2 was not taken out, but it could be if it were wanted. As to selective adsorption and the coating of the charcoal with oil, he would refer to the Dessau plant in that connection. The whole of the charcoal was coated with lubricating oil, and there was no adsorption at all.

If the charcoal had been coated with cresol the result would have been the same. He could not see why the coating of the charcoal with any oil would improve the adsorptive capacity. Mr. Parrish had raised the question of H_2S and the failure of the French process. The Baeyer Co. realised what had happened there, but after going into the matter very carefully they had decided to put up a big plant for $3\frac{1}{2}$ million cubic feet. The figures of the working of this plant should be available shortly. The figures obtained from the small plant at Leverkusen led the company to consider that it was reasonable to put down an ammonium sulphide extraction plant at Hüls. A chlorbenzol extraction plant had also been put down, and the company was going

to work the two in order to ascertain which was the most economical process. When the figures were available he would publish them. He agreed with Mr. Sutcliffe that a great deal had been done in this country in connection with the manufacture of carbon, but he was sorry that there was no activated carbon plant working in this country. With regard to the control of the alcohol plant, mentioned by Mr. Eric Reavell, the control was by smell. The alcohol could be smelt coming through at the break point, and there was practically no other test. The actual measured input into the plant was over 95 per cent. With regard to clearing the naphthalene filters at Leverkusen, the plant had been working for 24 weeks, and the filters had been taken out three times and burned in a retort with consequent losses of charcoal. There was a loss of about 10 per cent. the first time, 5 per cent. the second time, and 20 per cent. the third time. Now they were going to wash the filters out with benzol and recover the benzol by steaming it out, so that all the benzol would be recovered. The temperature of the gases, also mentioned by Mr. Reavell, had a certain effect. If there was a higher temperature, there was a lower adsorption, but it had been found possible to put gases through at 100 C. with complete debenzolisation. As to the efficiency of recovery, the answer was that the recovery was complete. Everything was taken out except the water. It could certainly be said that there is a 95 per cent. recovery, but actually he claimed the same efficiency as Prof. Williams did for the silica gel process — viz., 97 per cent. for the alcohol and 100 per cent. for the benzol. The recovery at Leverkusen was not complete because the plant had only been worked intermittently. At Dessau it was up to 100 per cent. He had not the figures for Bernau. The question of the efficiency of recovery there, however, did not come in because they were working under town gas conditions and they did not wish for complete removal. It was only desired to get about 1½ gallons out of the two gallons in the gas. With proper filtered gas 100 per cent. recovery could be obtained. With regard to benzol recovery and rectification, the benzol was steamed out and was practically water white. It would not pass the N.B.A. specification at present, but it was being used in Germany. It could be distilled and a practically pure product obtained, but even that would not pass the N.B.A. specification. With regard to the life of the carbon, plants had been working for 15 or 16 months, and there was no apparent loss in the activity. The mechanical ease was certainly one of the most pleasing aspects of the static process. He did not think it was possible to get anything simpler than the ordinary static plant. There were no mechanical parts, no pumps except for the products : in some cases it was necessary to have gas boosters, and these were practically the only mechanical parts in the plant.

On the whole, he did not think there could be anything simpler than the filter type of plant he had described.

Prof. Williams, in the course of his final reply, said that there is no price for silica gel, but he did not think there was any harm in giving Mr. Potter the figure of royalty he had asked for. It was 1·37d. per gallon of refined spirit made by the process. He was very glad that Mr. Parrish had raised the point with regard to the two processes not being in competition. That was the last thing he wanted to do. It was a great pleasure to him that Major Gloag had read his paper at the same time, because it had enabled a much better discussion to take place, but he did not put the two processes in competition in any way. As to the amount of gas required to force the gel through the tubes, the whole of the gas being treated was forced through. In other words, the gas pushed through the tubes was the gas from which they were adsorbing.

Prof. Williams said with regard to mechanical ease of the plant, if they looked at a silica gel plant it might seem a little complicated, but it was a great advantage for the chemical engineer to have a process which was suitable for continuous use, even though from the purely mechanical point of view it might seem a little complicated. By mechanical ease—the term used in his paper—he had meant ease of operation and output, and, as an instance of that, he had taken out one or two figures roughly to give some idea of what was gained by continuous working on a large scale. The gel in a silica gel plant went round, roughly, in 90 minutes and did 16 cycles a day. In doing that it adsorbed 128 per cent. with 8 per cent. adsorption of its own weight of benzol. That was admittedly not so high in adsorbtive power as carbon, and therefore he was giving the carbon process an advantage when compared with the percentage recovery in 24 hours given in Major Gloag's paper. At the same time the actual adsorption by the activated carbon process was only 25 per cent., as against the 128 per cent. with silica gel, taking the whole 24 hours working, so that from that point of view the silica gel was more adsorptive.

Major Gloag : But we have four cycles a day.

Prof. Williams said that the number of cycles did not come into this comparison. It was the amount adsorbed in 24 hours which he was comparing, and he claimed that one gets a bigger result with silica gel than with activated carbon. With regard to the colour of benzol, the National Benzole Association had published a lengthy report of his own, and copies were available for those who desired to have them. He entirely agreed that the test mentioned by Dr. Ormandy was of no value, and the N.B.A. was aiming to get it abolished.

A hearty vote of thanks was accorded the authors at the conclusion of the papers.

KINETIC ELUTRIATION

By LEONARD ANDREWS, M.Inst.C.E., M.I.E.E.

Elutriation, or the hydraulic classification of powders, is one of those many branches of physical science which attract one in the first instance by their alluring simplicity, and subsequently induce persistent research by the frequent occurrence of unexpected phenomena due to causes not originally appreciated.

What could at first sight be simpler than the well-known Stokes law :—

$$V = \frac{2}{9} \, ga^2 \, \frac{S-s}{\mu} \text{ in c.g.s. units,}$$

where a = radius and S = sp. gr. of particle, s = sp. gr. and μ = viscosity of fluid, giving the ultimate velocity of small particles of matter falling freely in a fluid ? For practical purposes it may be said that the velocity acquired by particles of a powder of a given sp. gr. falling freely in water is approximately proportional to the square of their diameters. If, for example, we take two specimens of, say, Fullers earth previously graded into sizes approximately 0·02 mm. and 0·04 mm. diam. respectively, and, after mixing with water, allow these to fall freely in a tank or beaker of standing water, we find that the larger particles acquire a velocity of approximately 1·2mm. per sec., whereas the velocity of the smaller particles does not exceed 0·3 mm. per sec.

If we now take a tank, constructed to provide an upward flow of water at any desired velocity, with provision for admission of the powders midway in the stream (Fig. 1), and allow the water to flow up through the tank at a velocity of, say, 0·6mm. per sec., the larger particles will descend against the rising water at a velocity of (1·2—0·6)=0·6mm. per second, and the smaller particles will be carried up and out of the vessel with the overflow at a velocity of (0·6—0·3)=0·3 mm. per second. This is an example of classification by gravitational elutriation in its simplest form.

Before discussing the design of elutriators for industrial purposes it is necessary to consider very briefly what are the respective forces which cause particles of different sizes to fall through water at different velocities.

To appreciate the nature and effect of these forces upon powders, it is helpful to consider first the causes controlling the respective velocities of comparatively large bodies falling freely in vacuo and in water. Imagine two spherical bodies of 1 cm. and 0·5 cm. diam. respectively sp. gr. 2·65 suspended in an exhausted cylinder, so that when released they will fall through a distance of 800 cm. (26·2 ft.). We know that both bodies will reach the bottom in approximately 1¼ seconds and both will have

acquired a velocity of 1260 cm. per second (41·4 ft per sec.). The momentum or kinetic energy ($\frac{1}{2}$ MV²) acquired by the respective bodies falling at the same velocities is obviously directly proportional to their respective masses or to the cube of their diameters. As the weight of the bodies is 1·385 grammes and 0·175 grammes respectively, they will acquire kinetic energies of 1120 and 140 centimetregrammes in falling through 800 cm. If now the experiment is repeated with the cylinder filled with water, having a free surface, the terminal velocity of the 1 cm. sphere will be reduced to 413 cm. per sec. and the maximum kinetic energy acquired will be only 121
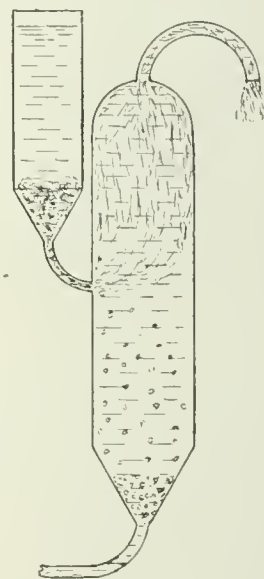
centimetre-grammes, whereas the terminal velocity of the 0·5 cm. sphere will be 290 cm. per second, and its maximum kinetic energy will be 7·5 centimetre-grammes. We have to account therefore, for a loss of energy of (1120—121)=999 centimetre-gramme units in the larger sphere and (140—7·5)=132·6 units in the smaller sphere when falling through water as compared to falling in vacuo. These losses may be visualised by plotting as in Fig. 2 velocity and energy curves of, say, the 0·5 cm. sphere when falling 800 cm. in vacuo and in water respectively.

The potential energy of the suspended sphere will be 140 centimetre-grammes (viz., its weight of 0·175 grammes × by the height of 800 cm. through which it may fall). The diagonal straight line A B shows the loss of potential and gain of kinetic

energy at any point of its fall in vacuo. As there are no losses when falling in vacuo the sum of the potential and kinetic energies will be constant at all points. The curve C D shows the velocity acquired at all points in vacuo, whereas CE. shows the corresponding velocity in water. The ordinates AB—AG represent the energy absorbed in lifting water of the same volume as the falling body through an equal height. In fact curve A G shows the kinetic energy gained by a 0·5 cm. sphere of sp. gr. 1·65 (S—s) falling in vacuo, and represents therefore the *effective weight* of the falling body in water. Curve A F represents the actual kinetic energy of the falling body. The balance of energy represented by ordinates AB—AF is absorbed by resistance.

Professor Perry found that below a certain critical speed the friction between fluid and solid was directly proportional to the velocity, and to the area of the wetted surface of the solid At the critical speed there is a sudden change in the law, and above that
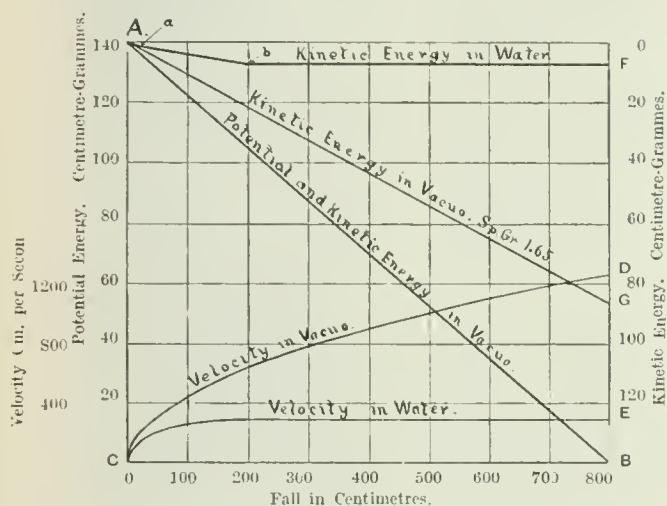
FIG. 2.

speed the friction is proportional to a higher power of the speed than 1.

The sudden change in the resistance at the critical speed is shown by the sharp kink at "a" in the kinetic energy curve (Fig. 2). From A to "a" this curve is practically coincident with the effective weight curve AG, the divergence shown being the result of the resistance due to viscosity which is directly proportional to the velocity. At "a" eddy currents are set up in the fluid which greatly increase the resistance, with the result that the rate of increase of kinetic energy is greatly reduced. The resistance now increases with the square of the velocity, until at "b" the loss of energy due to resistance exactly balances the gain of kinetic energy acquired per cm. by a 0·5 cm. sphere (sp. gr. 1·65) falling in vacuo. From this point onwards there can be no increase in kinetic energy to offset the loss of potential energy and the velocity, consequently, remains constant. This is, therefore, the ultimate terminal velocity upon which classification by gravitational elutriation is based.

In the case of fine powders falling in water the mass

of the particles is so small relatively to their wetted surfaces that the viscosity resistance alone is sufficient to prevent them reaching the critical velocity at which eddy currents are induced, with the result that the point of ultimate velocity "b" is reached before the point "a" at which the resistance increases in proportion to the square of the velocity. Fine powders in water fall therefore at velocities proportional to the sq. of their diameter, i.e., in accordance with Stokes law. Particles of sand larger than 0·2 mm. diameter induce eddy currents when falling in water, and their velocities vary approximately in proportion to the square root of their
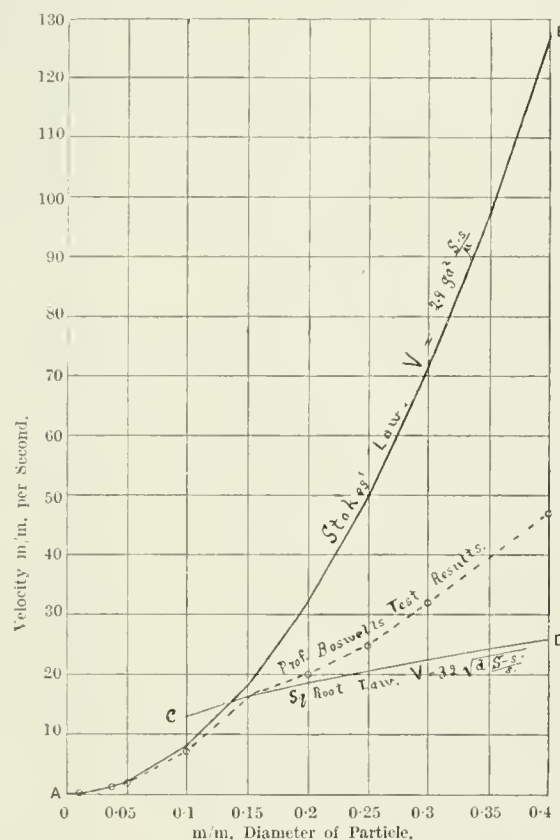

FIG. 3.

diameters. Curves AB and CD (Fig. 3) show respectively the theoretical velocities attained by particles of sand ranging from 0·03 to 0·4 mm. diam. sp. gr. 2·65 falling in water, AB being calculated from Stokes law and CD from the square root law. Prof. Boswell (Trans. Faraday Soc., Vol. XVIII.) has given results of actual tests he has carried out with sand of the above sp. gr. and diameters which results are also indicated in Fig. 3. It will be seen that these results are reasonably in accordance with Stokes law up to particles of 0·1 mm. diam., but above 0·2 mm. diam. they are more nearly in agreement with the square root law, but do not actually agree with either. It is unfortunate that no test results are given for particles between 0·1 and 0·2 mm. diam. Such results would probably fall on the dotted curve connecting the circles.

There are several other factors which affect the resistance and consequently the velocity of bodies falling in water. Col. de Villamil (" A.B.C. of Hydrodynamics "), in commenting upon Dr. Froude's classical experiments, points out that the resistance between solid and fluid depends greatly upon the extent to which the solid is wetted, *i.e.*, upon the adhesion or interfacial tension between the solid and fluid. Munroe found that a large number of spheres falling *en masse* fall with only one-tenth of the velocity of free falling spheres. Prof. Perry found that the resistance decreased rapidly with rise of temperature. Prof. Lowry (Trans. Faraday Soc., Vol. XVIII.) also found that the size of particle carried by water flowing at a given velocity was increased by reducing the temperature of the water, but not to the extent that the viscosity of the water is increased by a corresponding reduction of temperature. Mr. Sulman has shown that interfacial tension is usually increased by reduction of temperature. The extent to which the particle is wetted would thereby be reduced, possibly resulting in a reduction of the volume of minute air bubbles adhering to the particles, and thus reducing their buoyancy.

In view of the widely varying forces to be considered it is practically impossible to predict, without experiment, the sizes of particles of different powders that will be carried by a stream of water of any given velocity. Fortunately, it is unnecessary to attempt to do so in connexion with industrial elutriation as the velocity may be adjusted until the desired maximum or minimum size of particle is obtained at each outlet of the plant. It is obviously important, however, that the design of the elutriator shall be such as to provide for adjustments of the velocities at different points in the apparatus without altering the velocities at other points.

If we take a spoonful of dry Bagshot sand, which consists of particles of various diameters ranging from 0·005 mm. to 0·3 mm., and allow this to fall into a beaker of standing water, it is reasonable to expect that the sand will descend through the water at widely different velocities proportional to the sq. of the diam. of the particles. To prevent the fine particles from floating on the surface, owing to the surface tension of the water, the spoon must be plunged below the surface before turning it over. It is reasonable to expect that the sand will descend through the water at widely different velocities proportional to the square of the diam. of the particles, the velocities ranging from 32 mm. per sec. for the 0·3 mm. particles down to the 0·05 mm. per sec. for the 0·005 mm. diam. particles. Actually we find that the entire contents of the spoon fall through the water at a speed considerably greater than the theoretical velocity of the largest particles alone.

If instead of dry Bagshot sand we take a spoonful of the same material previously mixed with water the results are the same. A mixture of 50 parts silver sand and 50 parts Fuller's earth is found to give the same results, which shows that this phenomenon is not due to some peculiar properties of the Bagshot sand.

If, however, we separate the larger particles of the sand from the latter mixture, taking only particles of approximately 0·4 mm. diam. (wet or dry), the contents of the spoon break up into a shower of individual particles and fall through the water at speeds proportional to the square root of their diameters. Clean sand of slightly smaller diam., approximately 0·3 mm., does not break up but falls in bulk retaining the contour of the bowl of the spoon.

As this aggregation or flocculation of fine particles has always been one of the greatest difficulties associated with elutriation, before discussing methods of deflocculation it will be helpful to endeavour to form a mental picture of the forces holding the particles together. It appears to be generally accepted that every solid particle of matter that is readily wetted by water, immediately upon its immersion therein, surrounds itself with a film of the fluid which it retains in its passage through the water and even upon its removal therefrom until the film is evaporated or the particle otherwise dried. It is suggested, therefore, that immediately the spoonful of Bagshot sand is immersed the particles in the outer layers instantly surround themselves with their respective films, which adhere to each other, forming a chain armour or envelope around all the internal grains of sand or powder, which envelope not only holds the particles together but prevents them from being wetted.

If we puncture the envelope of a spoonful of sand and powder that has been left standing in water for a considerable time it immediately contracts to the sides of the spoon, thereby showing that it has been in a state of tension, and the contents of the spoon are found to be perfectly dry and dusty.

The experiment demonstrating the aggregation of particles that had been mixed with water before immersion in the beaker appears to require a slightly different explanation. In this case the films surrounding the individual solid particles appear to be of a cellular or honeycomb formation and may be likened to the foam or collection of minute bubbles on, say, a glass of stout. The films forming the walls of the cells are in a state of tension, and are only prevented from contracting by the pressure of the gas in the case of the foam or by the solid particles in the case of sand or powders. These stretched films bring the solid particles into close proximity to each other and they are then further held together by the molecular attraction between the molecules of which the solids are composed. Physicists tell us that the maximum distance through which molecular forces exert their influence is probably less than 1/250,000 part of an inch.

It is difficult for practical engineers to visualize these exceedingly minute dimensions, but it is very necessary to do so as these molecular forces have an important bearing upon the design of industrial elutriation plants. If a truly spherical particle of powder, 0·065 mm. diam. (200 mesh), could be magnified 100,000 diams. it would appear to be approximately 22 ft. in diam. The corresponding diameter of an imaginary sphere, the radius of which represented the maximum distance of molecular attraction, enlarged to the same extent

(100,000 diams.) would still be less than one inch. If, therefore, two of the above 0·065 mm. diam. particles are brought into intimate contact so that they exert the maximum attractive force upon each other, less than $\frac{1}{4}$ of a millionth of the molecules of which the respective particles are composed will come within the range of molecular attraction.

It appears that the intensity with which the particles in an aggregation of wetted powders are held together is mainly dependent upon the number of points of contact in a given volume.

In Fig. 4, " a " and " b " are respective micrographs of the sand which scattered when emptied from the spoon into a beaker of water and of the sand which sank in a mass retaining the contour of the spoon. The only apparent difference between them is that the average diam. of the " a " grains (which scatter) is about 0·4 mm. and that of the

a.   b.

Fig. 4.

" b " grains about 0·3 mm. The number of points of contact with true spheres closely packed on opposite sides of a plane of contact of given area is obviously inversely proportional to the square of the diameter of the spheres and directly proportional to the area of contact. It follows that the number of points of contact and hence the intensity of adhesion of the 0·3 mm. grains relatively to that of the 0·4 mm. grains is as 16 to 9 or nearly double. This would appear to be a possible explanation of the marked difference of behaviour on immersion of the spoonful of 0·4 mm. grains and that of the 0·3 mm. grains. It is conceivable that the smaller number of points of contact between the former so reduces their adhesive force that they are unable to resist the disruptive action of the eddy currents induced by their passage through the water, with the result that the aggregation is broken up and the particles scattered. In the case of fine powders the number of points of contact is of course enormously greater : thus if the aggregation consists of powders

averaging 0·01 mm. diam., the number of points of contact in a given area of contact surface is 1600 times greater than that of sand averaging 0·4 mm. diam.

Prof. Boys has shown that the pressure exerted by a bubble film upon its contents varies directly as the curvature of the film. As the curvature of the films surrounding small particles is greater than that of the films round larger particles, the force bringing the particles within the sphere of molecular influence would presumably be correspondingly greater the smaller the particle. Mr. Sulman also attributes flocculation to the particles being pulled together by the strained layers of liquid which surround them, when their own adhesive forces come into play.

Whatever may be the correct physical explanation of the forces producing and maintaining flocculation, the important consideration from a practical engineer's point of view is the minute dimensions and the enormous quantities of individual forces to be dealt with. Before classification of particles can be effected, every individual particle must by some means be so far separated from its neighbours as to be beyond the range of molecular attraction.

A number of ingenious mechanical agitators, rotary stirrers, rakes, etc., have been evolved for the above purpose. Such devices can, however, only dislodge an infinitesimal portion of the aggregated particles in a large mass of fine powders. Deflocculation by the addition of suitable electrolytes as demonstrated by Dr. Ormandy, with or without subsequent dehydration by electric osmosis appears to give much more satisfactory results. The only other alternative at present seems to be hydraulic erosion, combined with some means of imparting energy of motion to the aggregations and individual particles whereby they may be caused to bombard each other with sufficient force to remove the particles from the range of molecular attraction.

The chief drawback to gravitational elutriation is the extreme slowness of the process due to the very low velocities acquired by fine particles falling in water under the influence of gravity. Designers of centrifugal separators have demonstrated that the force of gravity may be multiplied many thousands of times by imparting mechanically a relatively high velocity to the solid particles. Centrifugal elutriators also have been used in laboratories for many years.

The term " kinetic elutriation " is used in the present paper to distinguish classification by gravitational elutriation from processes in which the velocity acquired is derived from some external force. The latter may be increased, obviously, to any desired extent, whereas the available force in gravitational elutriation is dependent solely upon the effective weight of the particle less the friction between solid and fluid. In centrifugal separators velocity is communicated to the solid particles by rotating the containing vessel at a high speed, but in kinetic elutriation any desired velocity may be imparted to the particles in a standing vessel by admitting water under pressure through a nozzle. Fig. 5 is a diagrammatic illustration of a simple but inefficient kinetic elutriator. In this case the solid particles
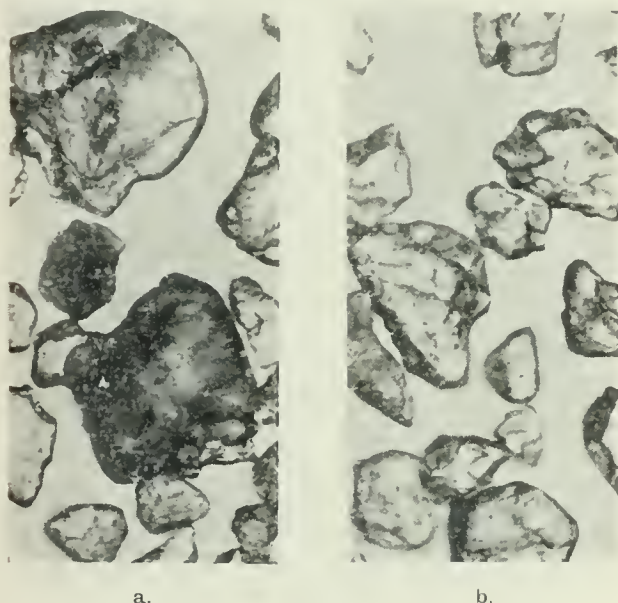
being mixed with the water before it passes through the nozzle, all acquire practically the same velocity, but the greater momentum of the larger particles enables them to overcome the resistance of the fluid and the force of gravity through a greater distance than the smaller and lighter particles.

Another construction, illustrated in Fig. 6 gives a reverse distribution. In this case the velocity acquired by the respective particles is directly
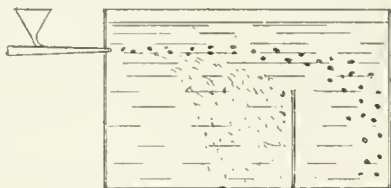

FIG. 5.

proportional to the time during which they are submitted to the external force and inversely proportional to their masses. As the larger particles fall much more rapidly than the smaller ones they will be acted upon by the high pressure stream for a much shorter time, and will consequently fall into the nearest receptacle, whereas the fine particles only will be carried over the barrier into the second receptacle. One of the most interesting and instructive examples of kinetic elutriation is the remarkably even grading of small cobblestones, shingle, gravel, sand and silt, by the action of the sea waves. There are unquestionably many lessons to be learnt, helpful in the design of industrial
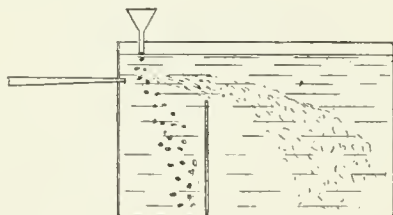

FIG. 6.

elutriation plants, by a study of the action of the sea waves. What, for instance, is the nature of the combination of forces which selects, say, cobblestones of striking uniformity and size, from the mass and great variety of material slowly drifting in the deep water of the English Channel, and deposits them on the highest portions of the beach, the smaller material being deposited at the same time, grade by grade, at lower levels? There appears to be much difference of opinion amongst experts who have made a life study of the complex forces of wave and tidal action, as to the relative work done by wind and tide in constantly shifting the material around the sea coast. It is quite obvious, however, that the classification referred to is not the result of gravitational elutriation.

It now only remains to consider how the general principles discussed can be best applied to industrial elutriation. After experimenting with a large number

of sectional models, the author has obtained the best results with the construction shown diagrammatically in Fig. 7.

Water under pressure is supplied intermittently through the automatic valve A and the multi-stage ejector BB[1] to the annular vortex chamber C, thereby imparting a rotational velocity to the water in this chamber. During the periods when the supply of water under pressure is interrupted the free water surfaces in the feed hopper D, the float tank E, and in the delivery pipe to the overflow tank F, will all stand at the same level, but immediately the automatic valve opens, the centrifugal force of the rotating water in the vortex chamber will raise the level of the water in the overflow tank, whereas the partial vacuum induced in the suction pipe of the first stage ejector B will tend to lower the level of water in the feed hopper.

The overflow tank F may be raised by the rack and pinion G until the increased static head of the water in the raised tank just balances the kinetic head resulting from the rotational velocity of the water in the vortex chamber, when the flow through the delivery pipe will cease, just as the flow through the delivery pipe of a centrifugal pump ceases when the height of overflow exceeds the critical kinetic head imparted by a given velocity of the impeller. If, however, solid material heavier than water is now allowed to flow into the elutriator from the feed tank, the density, and hence the mass of the rotating fluid in the vortex chamber, will be increased, thereby increasing the kinetic head and causing water again to flow into and out of the raised overflow tank.

The coarsest and heaviest particles of solid material will be discharged more readily by the centrifugal force of the fluid in the vortex chamber than the smaller particles in view of their greater mass and similar velocity, and these heavy particles will tend to gravitate to the bottom of hopper H. It is necessary to allow some water, and consequently some of the fine particles in suspension, to flow with the coarse material into H, but this water will flow up the delivery pipe to the overflow tank and be discharged therefrom into the float tank. To avoid intermediate coarse particles also being carried to the overflow tank an expansion chamber I is inserted in the delivery pipe which, by reducing the velocity of flow, limits the size of particles carried through it. During the half period that the supply of high pressure or motive water is discontinued the circulation of water in the vortex chamber ceases and the kinetic head falls to zero, with the result that the water accumulated in the overflow tank ebbs, returning through the expansion chamber until the levels in overflow tank and float tank are equal. This reversal of flow carries any intermediate coarse particles that may have accumulated in the bottom of the expansion chamber back into hopper H. The fine material is prevented from returning by so limiting the capacity of the overflow tank that only a small volume of water returns before equal levels are attained, when the return flow ceases. To prevent any of the fine particles passing through hopper H from descending into the final coarse hopper J, a small stream of water

is caused to flow upwards through the narrow channel interconnecting the two hoppers, which upward flowing stream also agitates the material settling at the bottom of H, effectually carrying up any fine particles and causing the heaviest particles to work to the bottom of the hopper. This auxiliary stream is supplied through a small branch pipe from the main supply to the ejector and is therefore also intermittent. When the flow is interrupted the

material leaving the conveyor has been found to contain less than 25 per cent. of water.

To avoid a rush of water, tending to drain the entire apparatus when the coarse discharge gate is opened, a sand seal is maintained in hopper J, which is not allowed to fall below a predetermined level. This is effected by means of an electric feeler shown diagrammatically in Fig. 8. Two arms of an electric switch are free to move about a
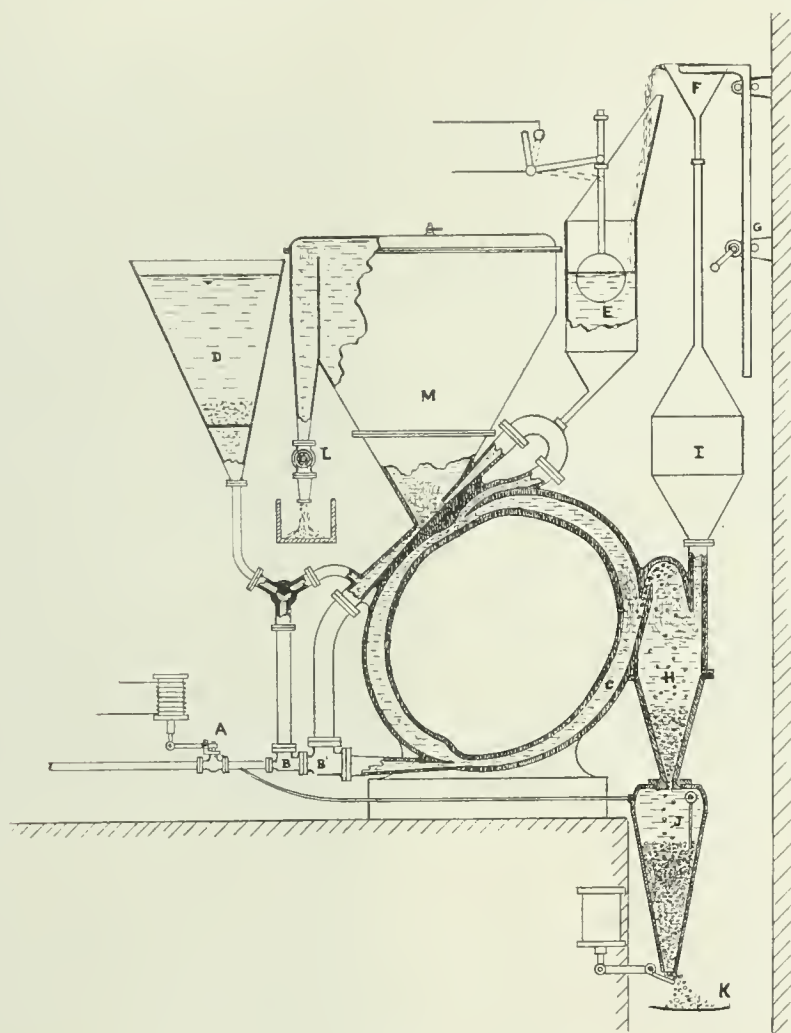
Fig. 7.

coarsest and cleanest material gravitates into the final hopper. It has been stated that there are practically no hydraulic classifiers which do not allow some of the fine material to escape with the coarse. It can be demonstrated however that the coarse material descending into hopper J is entirely free from fine particles as is evidenced by the clearness of the water in J in marked contrast to that in H, which is densely crowded with suspended matter. The coarse material is discharged from J through an electrically operated gate on to a belt conveyor K by means of which it is drained of surplus water and is returned to the feed of the grinding mill. The

common fulcrum. One of these arms, which is provided with an insulated contact stud, is mechanically connected to the automatic valve controlling the water supply to the ejectors and it consequently rocks backwards and forwards every time the valve is opened and closed. The second switch arm, which is weighted, rests on a stop on the first arm and consequently tends to move with it. This second arm carries a blade on the end of a spindle projecting into hopper J, which, so long as the coarse material is below the predetermined level, also swings backwards and forwards with the switch arms. As soon as the material rises sufficiently

to anchor the blade the first switch arm in its move-
ment does not carry the second arm with it, the
contacts are consequently brought together and the
electric circuit is completed through the solenoid
operating the discharge gate. When sufficient
material has run out to release the blade the
electric circuit is again broken and the discharge
gate closed.

Returning now to the vortex chamber it is obvious
that only a small proportion—and that the largest
and cleanest coarse particles—will be discharged by
centrifugal force into hopper H, the balance, including
the bulk of the fine material and aggregations of
coarse and fine, will be carried on by the viscosity
of the water and circulated round and round the
vortex chamber. Since a considerably greater volume
of water is flowing in than is discharged into H, the
surplus water must escape through some other outlet.
This second outlet is connected tangentially to the
top of the vortex chamber in a reverse direction to
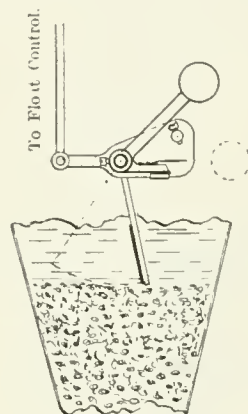the vortex flow. The momentum of the coarse

FIG. 8.

particles in the vortex stream carries them past the
latter outlet, but the fine particles, with their much
greater surface area, relatively to their mass, are
carried by the viscosity of the outward flowing
stream into the bottom of the syphon chamber M
through a nozzle—thus gaining velocity. The
syphon chamber is provided with an outlet directly
opposite the inlet nozzle, which outlet is connected
to the suction inlet of the second stage ejector B¹,
with the result that any coarse or intermediate
particles that may have been carried up with
the fine material are projected by their mo-
mentum directly across the bottom of the
syphon chamber. The area of the outlet
relatively to the inlet nozzle is so proportioned
that the velocity at which the water leaves the
syphon chamber is very much lower than that at
which it enters, and the impact of the high velocity
water upon the low induces eddy currents or a
rotational velocity which carries the fine material
up into the syphon stream but is insufficient to
divert the coarse particles from their direct course.
The velocity of the syphon stream is controlled by
the valve L. This flow, which is continuous and
entirely unaffected by the intermittent velocities in

other parts of the system, determines both directly
and indirectly the maximum size of the particles
carried by the main overflow.

The connection between the coarse outlet from
syphon chamber and suction inlet of the second stage
ejector B¹ is branched to communicate, through a
3-way valve, with the suction inlet of the first stage
ejector B, and with the feed hopper. The position
of this 3-way valve regulates the rate of flow of solid
material relatively to the flow of water. The feed
hopper is provided with a 20-mesh sieve which, in
addition to holding up any coarse material etc.
exceeding 0·65 mm. diam. breaks up the flocculated
material into aggregations not exceeding 0·65 mm.
diam. These aggregations are further subdivided
by hydraulic erosion and by the repeated impact
with each other caused by the many changes of
direction and velocity in their passage through the
system. As previously mentioned, water in the
feed hopper ebbs and flows with the intermittent
action of the ejectors. When flowing into the hopper
it lifts the flocculated solid material off the sieve and
thus clears the mesh which without this action very
quickly clogs. When flowing from the hopper it
forces the solid material through the sieve.

As water is admitted to the system intermittently,
but flows out of the syphon chamber continuously
at a constant rate, it is obvious that the level of the
water in the float tank will rise when the automatic
valve controlling the supply to the ejectors is opened,
and will fall when this valve is closed. The float is
arranged to control mechanically or electrically the
automatic valve A, which is thereby respectively
opened and closed at predetermined low- and high-
level limits of the water. As both high- and low-
level limits may be adjusted as desired, the periodicity
of the ebb and flow upon which so much depends
may be varied to any required extent.

In cases where it is desired to separate the material
into 3, 4 or more grades, as is necessary for ore
dressing, the contruction shown in Fig. 7 is practi-
cally duplicated or triplicated with the exception that
the syphon chamber is only required for the final
stage. In earlier stages, instead of the syphon
chamber, an interconnecting pipe is carried to the
feed inlet of the next stage. The arrangement is
somewhat equivalent to that of a multi-stage turbine
pump. As additional water is added at each stage
and utilized for imparting velocity to the fluid in
each respective vortex chamber the volume and
consequently the velocity is gradually increased as
the mass of the individual particles to be discharged
into the respective coarse hoppers becomes succes-
sively smaller.

The micrographs reproduced in Fig. 9, all en-
larged 50 diameters, show results obtained in grading
powders etc. in a 2-stage elutriator, A being material
as fed in (0·005 to 0·4 mm.), B first coarse discharge
(0·1 to 0·4 mm.), D second coarse discharge (0·02 to
0·2 mm.), and C fine material (0·005 to 0·03 mm.).

The fine particles ultimately discharged with the
overflow will be suspended in from 10 to 20 times
their own weight of water. The bulk of this water
may be separated by running the overflow into large
settling tanks if time and space are no object, but
in most cases it will be preferable to extract the

water, either by a centrifugal separator, a streamline filter or an electric osmose machine. In the event of a grading centrifuge being used for this purpose, such as the " Gee " Centrifugal Separator, it will probably be advantageous to treat the Kinetic Elutriator as a process for breaking up the flocculations, and for extracting material of diameters ranging from, say, 0·65 mm. down to 0·04 mm., effecting the final classification simultaneously with the process of dehydration. The high velocity necessary for efficient dehydration is, in fact, as beneficial for classifying very fine material as it is detrimental for separating the coarser particles from the fine.

It is unnecessary to enlarge upon the many applications of industrial elutriation to the chemical industries, as these must be obvious. Whilst for some materials some form of dry grading is imperative, there are many more where the material has been

have formulated laws for the guidance of practical designers without which the progress already made in industrial elutriation would have been impossible.

DISCUSSION.

Mr. W. J. GEE said the author had certainly given a new view-point upon the problem of elutriation, and had also given a stimulus for further consideration of it. The paper started with a reference to Stokes' law, and far be it from him to attempt to belittle Prof. Stokes or throw doubt upon his law, which was absolutely right, but there was no doubt that it applied to conditions that are never met with in actual practice. If we could imagine a perfectly quiescent liquid, then the particles would behave exactly as Prof. Stokes said they would, but the commercial conditions came into play, and the engineer wanted to know within what percentage

A.   B.   C.   D.
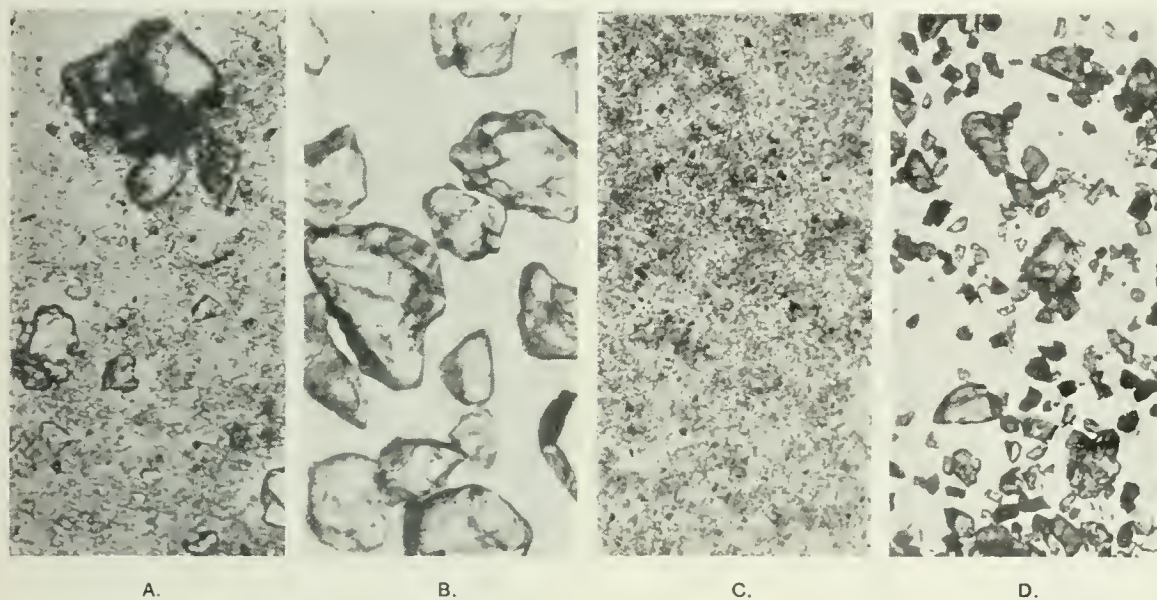
Fig. 9.

already wetted for grinding or other processes in connexion with which grading by elutriation may be used advantageously. In Mining Industries this method of grading has been used extensively for many years, and there are probably few, if any, large ore dressing plants that do not contain a number of hydraulic classifiers, although according to some authorities on ore dressing the work done by many of these somewhat crude classifiers is very imperfect.

In Civil Engineering the treatment of sewage and the purification of water appear to present considerable opportunities for the application of the general principles of elutriation. In coal-washing appliances and in the treatment of china clay and other pottery materials the principles have already been largely applied.

In conclusion, the author wishes to take this opportunity of remarking how much he has had cause to appreciate the indebtedness of engineers to those several physicists who have closely studied for many years the properties of powders in suspension, and

any particular apparatus would work. Would it work on a 5 per cent. suspension or a 10 per cent. suspension, or must it be only a 2 per cent. suspension ? The result was that instead of there being one size particle settling in water, there were countless numbers of them, and the difficulty was that the particles collided with one another and the heavier ones brought down the light ones by entanglement, which set up the trouble that the author had referred to of flocculation and aggregation of particles. He was glad the author had dealt so fully with flocculation because that was undoubtedly one of the great difficulties in elutriation, particularly when dealing with heavy suspensions. The author had spoken of elutriation as the hydraulic classification of powders, but elutriation really had two sides. There was the classification of powders as regards size—which presumably was the kind of elutriation which the author had in mind—and there was also the much more interesting and much more difficult problem of the separation of particles according to

their specific gravity. It was quite easy to size particles if they were all of the same specific gravity ; similarly, it was not difficult to separate the heavier particles from the light ones if the particles were of the same size and different specific gravity, but perhaps the plague of the matter was when all these four things came together. The tendency was for the heavier stuff to go down with the coarse and for the light stuff to go down with the fine. He hoped the author would be able to give some assistance in this problem when he had gone a little further in his investigations. What was required was a sizing apparatus which will size whatever the specific gravity of the particles, and, further, a specific gravity separator was required which will separate according to specific gravity no matter what the size of the particles might be. It might be possible for the author to adapt his apparatus so that it would do what is required in two stages ; first, a specific gravity separation, and then a separation to get the particles of the same size or nearly the same size. Would the author say what percentage suspension he has been able to work in his apparatus, and at what point did he find the classification interfered with so badly that it did not have any effect ? There was bound to be some limiting factor of this sort. He would also like to know whether the author had done anything as regards separation according to specific gravity, and further, if it were not too soon, had the author considered the question of the cost of the apparatus and the cost of working it as compared with any of the existing plants ?

Mr. J. McConnell Sanders said that as a chemist he was a little inclined to criticise the fact that the author had dealt with this problem almost entirely from the physical and engineering point of view. By a curious coincidence the author had chosen for the purpose of his experiments that evening three substances which in the practice of the petroleum industry had shown themselves to be more than simply physically inert substances, viz., fuller's earth, sand and silica gel. These all showed that curious phenomenon known as adsorption, and it seemed to him that substances of that kind must necessarily be affected by the nature of the liquid in which they are suspended. The chemical composition of the water must play a part, particularly in the case of sea water. Again, in connexion with the micro-photographs, shown by the author of particles which scatter and particles which do not, it was stated that there was no other difference than that of size, but it seemed to him that the particles which scattered most were round, smooth particles, whilst the particles which did not scatter were sharp-edged. Supposing there was anything in the theory that particles suspended in a liquid are imbued with electrical charges, was there not a possibility that the ease with which these discharges could be dispersed from a sharp-pointed particle might have something to do with the ease with which they are formed ? The particle with a sharp surface might be presumed to have a greater amount of surface energy. Again, the phenomenon known as adsorption, in which colouring matter—unsaturated hydrocarbons and sulphur compounds—are taken out

of an oil simply by some physical condition of the surface of the material from which they are filtered, might have something to do with the ease with which particles would fall in a liquid. The author's apparatus seemed a little complicated, but perhaps that was due to the fact that he had endeavoured to make a demonstration model. Was it not possible, however, that the large solid particles could be separated from the liquid without the necessary complication of using high-pressure water to impart velocity, by a combination of existing means of separating large particles from liquid ? The author in his apparatus had made use of a simple form of gravitational elutriation, with one of the oldest forms of segregating one size particle from another, viz., the common sieve, and it was for this reason he suggested the same result might be obtained without the use of high-pressure water.

Prof. J. W. Hinchley also thought the apparatus was a little complicated, perhaps due to the fact that the author had taken several bites at the same cherry. The fact remained, however, that he had done the job, which was quite sufficient for the moment, and he hoped later that the apparatus would be seen working. He did not agree with the author that the easiest way to study this problem is from the energy point of view, and what happened was quite clearly seen if one kept to the momentum point of view, rather than velocity. Mining men had studied this thing very carefully and could give quite a lot of information about the action of jigs. If one shook coarse powders, the coarse will come to the top, but if you shook them through pulsating water, the coarse will go to the bottom and the fine to the top, and he was inclined to think that in his little chamber, H, the author was doing a bit of jigging. With regard to the author's experiment concerning the interfacial tension arising between particles of sand, one was struck more by the examination of what happened with regard to petroleum or petrol. The creep of petrol along a surface was one of those curious things which still demand a lot of attention and were not yet clearly explained. When one delved into the physics of these fine particles, one was faced with a much more difficult problem than was imagined, and he did not think it had been completely explained by the author. It was necessary to go still farther and examine the actual character of the surface and not merely whether it was easily wetted. There was something else besides.

Mr. W. S. Lockhart said his experience had principally been in the separation of particles by specific gravity, and the conclusion he had come to was that one must make up one's mind what one wanted to do, and within a certain range attempt to do that and not to attempt to do too much at one time. The sine qua non of separation by hydraulic currents was to have a solid current. Wherever one used a current and had too small pipes or high pressure or anything of that sort, the current was destroyed altogether. There must be a vertical solid current without any eddies whatever, and that was where his experience and Mr. Andrews' views would clash. Mr. Andrews was working with eddies, whereas he himself was working with eddy-less

currents. Again Mr. Andrews was working with a high pressure—a head of 40 feet—whereas he himself was working with say tin, with a specific gravity of 6·5 or quartz with a specific gravity of 2·65. The speed of separation, of course, depended upon the size of the particle, but working with ¼ inch or ⅜ inch particles he found it was easy to separate these two materials with a head of water of ¼ inch, and he got an absolutely clean separation. He had recently been doing some work on coal and had utilised a 5/16th inch head of water, the difference of specific gravity being from 1·5 to 1·8. That coal was turned with under 1 per cent. of shale matter in it and the dirt was quite free from all coal or fuel matter. Thus his experience had been with low-water pressures, in order to avoid eddies which latter prevented any separation at all.

Mr. B. W. HOLMAN said it was a point of interest that Mr. Andrews had experimented with sands, the great mass of which had angular particles and did not correspond to spheres as Stokes' law assumed, and it would be interesting to know whether the introduction of the kinetic factor in this apparatus rendered a deviation from Stokes' law of less importance because the particles were angular. In other words, was the viscous resistance less influenced in these circumstances when using a kinetic elutriator than when using ordinary methods of classification with comparatively low heads? That was a point of considerable interest in connexion with certain ores with extremely fine particles disseminated in them. As a mining man he was not acquainted with the term elutriation to connote a commercial process. Apparently in chemical engineering elutriation was a term used for the separation of particles which the mining man would call classification, and this led to the question whether the author hoped to develop his apparatus into one for the treatment of ores and similar substances at the rate of many tons a day. Then there was the use of the terms "coarse" and "fine." Somebody had mentioned particles having a size of a fraction of an inch as being "coarse," and "fine" had been used to connote material which was almost of colloid dimensions, and it had been said that it was difficult to photograph the particles because they would not keep still. He would like to know the dimensions of the "fine" particles, and might it not be that the fineness of the particles was the reason why they would not keep still? Further, he would like to know whether what appeared to be comparatively low velocities compared with velocities in a centrifuge rendered possible the separation of colloid particles.

Mr. ANDREWS, replying to the discussion, said that the density of material which he could deal with in the apparatus was about one part of solid to 20 parts of water. If there were more than that it seemed to destroy the classification. The particles must have room to move about. There was no getting away from that. He could not say anything at the moment with regard to what the cost would be for an apparatus to deal with, say, 50 tons of material per day, but as an engineer he had endeavoured to keep the question of cost constantly in mind. One or two speakers had said that the apparatus was complicated, but there was no complication in design as such. There was a moving part and a weight falling backwards and forwards at the top, but the only real complication was the twisting channels of water. He agreed that Stokes' law is delightfully simple, but it did not carry us very far. Mr. Lockhart had spoken about eddies, and he fully recognised that to get really satisfactory elutriation there must be a solid current with no eddies. That was an old criticism of the Schoene tube, but that absence of eddies could not be worked with a plant intended for a few hundred tons a day.

Mr. LOCKHART said he would like to explain what he meant by a solid current. When the material was introduced an obstruction was introduced and the arrangements must be such that whatever obstruction was introduced the liquid would continue to flow at the original pace before the material was introduced. There must be no acceleration.

Mr. ANDREWS said he appreciated the point as to the effect of the obstruction, and for that reason he had introduced his material through a very small nozzle so that it all travelled at the same speed forward. There was no question of the water passing the obstruction; it picked it up and sucked it in. With regard to Prof. Hinchley's remarks, he personally looked upon kinetic energy and momentum as the same thing. What he could not think in was velocity as such. With regard to the last speaker, the dimensions of the particles on the slide to which reference had been made was a maximum of 20 microns. No doubt a great many of the particles were colloidal, but he could not tell what the "fines" really were. The movement was partly due to local vibration. In the case of the smaller particles there would, of course, be the Brownian movement, but that would not appear in the photographic slides.

A hearty vote of thanks was accorded the author at the conclusion of the discussion.

THE PREMIER AND OTHER DISINTEGRATING MILLS AND THEIR APPLICATIONS IN INDUSTRY

By F. J. C. CHINA

During the past few years considerable attention has been devoted to obtaining machinery which can disintegrate certain materials to a fine state of subdivision, very much finer than can be obtained by means of ordinary grinding machinery.

Many unsuccessful attempts have been made, but in most cases this is largely due to the fact that experimenters have been over-ambitious. The methods of grinding which have been in existence for centuries are all dependent upon actual attrition, and possibly the most accurate and best instrument which we have at our disposal for this work is a high grade roller mill such as is used in paint works. The rollers in a mill of this type can be made to within very fine limits, and we cannot conceive of
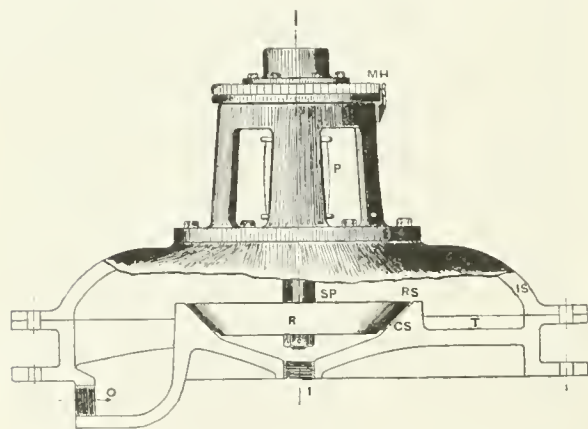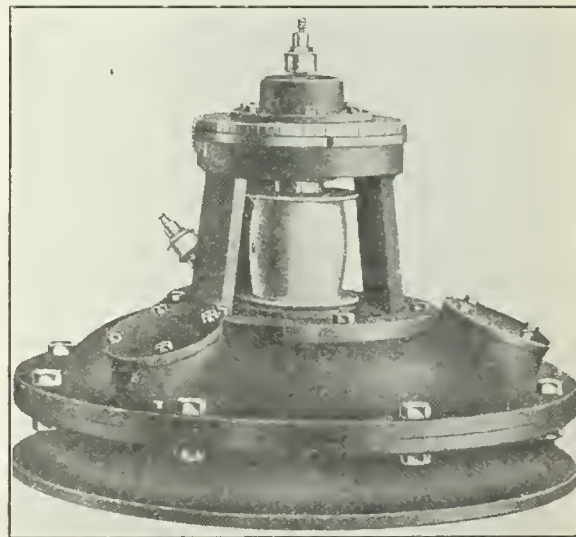

Fig. 1

anything very much better for the purpose. The limit of fineness obtained with this machinery varies enormously and in all cases depends upon the material under treatment.

Most of the recent attempts at obtaining very fine grinding of solid particles have had as their basis the utilisation of hydraulic forces. In other words, an attempt has been made to create enormous frictional resistance in a liquid in which is suspended the material required to be disintegrated. The resistance to disruption offered by a liquid subjected to rapid beating or shearing varies as the speed of attack and up to the present time it has not been possible to obtain sufficiently powerful hydraulic

forces to ensure disintegration of really hard material. This is largely dependent upon the fact that we cannot obtain high enough speed of rotating members in any particular type of mill.

The two chief types of mill which have been developed during the last few years are :—(1) the beater type, (2) the type in which a film of liquid is subjected to a shearing action at very high speed. The beater type of mill has had a certain amount of success, but has limitations which need not be discussed here as we are mainly concerned with mills of the latter type.

View of " Premier " Mill
Fig. 2

The Premier Mill, which falls under the second heading is typical of this kind of machine and will be described in detail, as the author has more experience of its working than of other similar mills. It consists of a cone which rotates in very close proximity to a conical seating and a glance at the diagram will show its general form.

Reference to the diagram (Fig. 1) shows that the mill consists of an outer casing which surrounds the rotor, R. This rotor consists of a perfectly

smooth steel cone which is made to extremely fine limits as regards accuracy, and fixed to the spindle, SP, which is mounted in special bearings. This cone works in close relation to a fixed surface, CS, which forms part of the casing, the arrangement being very similar to the action of the clutch on a motor car, except that the two surfaces do not touch.

The working surface of the rotor, RS, is arranged so that there is a very fine clearance between same and the surface of the casing, CS.

With this arrangement we therefore have a metal surface rotating in very close proximity to a fixed surface at a speed which may vary between 1000 and 5000 revolutions per minute.

The rotor is driven by the pulley, P, mounted on the spindle, SP.

The bearings which control this spindle are mounted in the micrometer head, MH, and in the top casing of the machine immediately below the pulley, P. The micrometer head enables the clearance between the rotor, RS, and the cone, CS, to be adjusted to any degree desired.

The rotor should never be adjusted so that it actually touches the casing, but it can be so regulated that the actual clearance can be anything from three-thousandths of an inch to two or three hundredths, according to the nature of the work to be done.

It is essential to realise that when the machine is being used with hot material, one must take into consideration the fact that the metal expands on heating. A fine clearance should therefore not be set until the machine is thoroughly heated up. It is a golden rule never to use a finer clearance nor a higher speed than is necessary for the job.

When operating the mill, the material to be dealt with is fed through the inlet I at a suitable rate, either by pump or gravitation. Very little head is required when feeding under the latter condition, because the rotor acts as a centrifugal pump to a large extent, and assists by drawing the material to be dealt with between the working surfaces. It then passes through the narrow space between the conical surfaces in a fine film and, owing to the enormous speed of the rotor, the particles of solids suspended in the film are rapidly torn apart.

Some typical applications of this type of mill are as follows :—

(i.) The perfect emulsification of liquids.
(ii.) The intensive mixing of liquids and solids and the blending of difficultly miscible constituents in various chemical preparations.
(iii.) The minute disintegration of solids suspended in liquids.
(iv.) The extraction of oils, e.g., the extraction of tar acids from tar oil, pyridine from tar oil, etc.
(v.) The extraction of fibrous tissues, e.g., complete extraction of meat tissues in the cold, and the extraction of vegetable or animal fibres of any nature.

(vi.) The rapid and economical manufacture of paints and enamels and liquid suspensions of all kinds of pigments.
(vii.) The regeneration of milk from milk powders, and the production of synthetic cream from butter and milk powder.
(viii.) The rendering of certain metallic ores into a flocculent suspension.
(ix.) The wet grinding of organic chemicals, dyestuffs, etc.
(x.) The manufacture of tannin extracts.
(xi.) The manufacture of pharmaceutical mixtures and emulsions of all kinds.

Taking these applications in order it would be as well to give a few typical cases.

(i.) *Emulsification.*—For many years emulsions have been made in various industries, but in nearly all cases it has only been possible to make such emulsions because the oils have either lent themselves to easy emulsification, or, as in the case of certain pharmaceutical preparations, have required large amounts of stabiliser for the purpose. After much experimental work it has been found possible to produce almost any kind of emulsion in a Colloid Mill providing one has the right stabiliser and the right conditions. Furthermore, it is very seldom necessary to use more than 2 or 3 per cent. of stabiliser and very often only a fraction of this quantity is necessary.

In going into these practical applications the writer makes no apology for not entering into any theoretical controversies, and only the practical points will be considered in this paper.

Depending on the conditions of manufacture an emulsion may consist of the oil in water type, or the water in oil type. As a general rule it will be found that most emulsions which enter into industry are of the oil in water type. Occasionally one is able to make an emulsion which falls outside this classification, that is to say, one may be able to dilute it with water or strengthen it up by adding more oil. As a general rule one does not consider an emulsion satisfactory unless it can be boiled without separation. Furthermore, one must be able to filter it and occasionally the emulsion will have to withstand freezing.

One of the first successful applications of the Premier Mill to an industrial emulsion process was that of impregnating timber with an emulsion of creosote oil in place of using neat creosote. Considerable work has been done on creosote emulsion with a view to impregnating wood with creosote in a dilute form, but so far as is known no emulsion has yet been prepared which will fulfil the following conditions :—

(1) The emulsion must be so stable that it may be boiled or frozen without breaking down.
(2) The degree of dispersion of the emulsion must be so fine that no filtration takes place when the emulsion is forced into the pores of the wood.

(3) The emulsion when made must contain so small a quantity of added bodies that these bodies do not have a tendency to leave the creosote in the wood in such a state that it can be partially washed out by water. That is to say, that the insoluble properties of the creosote must in no way be impaired.

(4) The emulsion must remain so stable during the process of impregnating the timber that the unused portion of the emulsion must retain its original composition.

(5) The emulsion as made must contain so small a quantity of added bodies that it is commercially practicable.

We have approached the subject from a chemical standpoint, the idea being that if creosote could be diluted with an inert substance to three or four times its volume, we could then get the same penetration of oil into the wood as is given by any of the above processes, and yet have only $\frac{1}{3}$ or $\frac{1}{4}$ the amount of oil left on the fibres after the process is finished.

Moreover, it is undesirable to leave the fibres loaded with an inert substance, as this may prevent the preservative action of the oil. It must, therefore, be extracted afterwards, or it must be capable of seasoning out.

Water is the cheapest diluent which could possibly be used, and has the additional advantage that it can be left to season out of the wood of its own accord after impregnation.

The difficulty, however, arises from the fact that the oil and water will not mix in the ordinary course of events, and even if the creosote is finely divided throughout the medium so as to form an emulsion, it is still a matter of considerable difficulty to make the emulsion stable and to get the particles small enough to enter the pores of the wood without being filtered out.

With the aid, however, of the Premier type or other Colloid Mill and a carefully selected stabiliser, we have succeeded in obtaining creosote in an exceedingly fine state of subdivision in water, and also in preventing these small particles from again coalescing, even when the temperature is raised to somewhere near boiling point.

This emulsion has been given the name of "Plexosote," and by its means it is claimed that one can obtain the same results, both as regards penetration and preservation, as can be obtained by any known process of creosoting, but with considerably less oil, and at a lower cost.

By suitable dilution, it is possible to leave in the wood any amount of oil desired, the lower limit of which to secure preservation is still a matter for determination.

Another commercial application of an emulsion is that of an emulsion of bitumen and water. The dressing of roads with tar and bitumen and the coating of road material with tar and bitumen is a very extensive business, but is expensive owing to the fact that in nearly all cases the material has to be heated. In the case of the spraying of the road, the material must be applied hot, and in the case of coating the stone, the stone itself must be dried before it can be coated.

Now, a soft bitumen can readily be emulsified in the Premier Mill with the aid of a suitable stabiliser, in which form it exists as a chocolate brown liquid which is miscible with water. This liquid can be readily sprayed on to the road cold, and soaks into the surface rather more than does the ordinary hot tar or bitumen. On coming in contact with the stone, the emulsion breaks down and the water very quickly leaves the emulsion, leaving behind the bitumen coating.

In practice, the road is sprayed in the ordinary way, and sprinkled with grit or dust in exactly the same way as it would be in the case of tar or soft bitumen.

In the case of coating stone, it need only be freed from dust, and it does not matter whether the surface of the stone is damp or not. All that is required to coat the stone is a suitable rapid mixer.

Extensive tests are being carried out this summer with this material, and it is confidently anticipated that it will give satisfactory results. One can readily see that there will be considerable saving in labour, as no heating apparatus is required in the spraying, and no drying plant is required in the coating of the stone.

Emulsions in various colours for internal and external decorators' purposes and preservations are readily prepared, and cheap and efficient products which give uniform effect and which bring out the grain of the wood find a ready market, especially for bungalows, roofing shingles, floor boards and the like.

These products consist merely of an emulsion of creosote and water, and should be made by adding a little pigment in such a fine state of subdivision that it is readily taken up into the pores of the wood.

Emulsions consisting of water and oil, such as creosote or petroleum oils, pitch, asphalt, bitumen or petroleum residues, are used as dust layers, and these can be applied in a very simple manner. Similar emulsions are very useful for rendering porous stone waterproof, and even for waterproofing certain building felts.

Emulsions of waxes, such as paraffin wax, beeswax, can easily be made. The only precaution to take is that the temperature shall be kept well above the melting point of the waxes in question while the emulsions are being made. They should be cooled as they leave the mill.

In all these cases a very small quantity of stabiliser is required which seldom exceeds 3 per cent.

A somewhat extraordinary application of an emulsion process is that of emulsifying petrol. The writer would not suggest that this process is of any practical value at the moment, but it is highly probable that it will become useful when further work has been done in this direction.

In utilising an emulsion of petrol and water in an internal combustion engine, one is forced to utilise a stabiliser which is completely combustible and one has very little choice but to accept ammonium oleate for this purpose.

Utilising from 1 per cent. to 2 per cent. of this stabiliser and the necessary conditions, there is no difficulty in making an emulsion containing anything from 10 per cent. to 75 per cent. of water. All emulsions containing up to 50 per cent. of water

by weight are of the water in oil variety. Above this percentage the emulsion is a little unstable as far as its type is concerned, but above 60 per cent. reverts to the oil in water type.

An emulsion of 30 per cent. of water in petrol is really quite a good fuel, and during this year a Chevrolet four-cylinder car has been run many hundreds of miles on it. A controversy as to the exact action of such a fuel is necessarily a lengthy one, and we should only say here that this fuel is thoroughly satisfactory for an engine of this type. If the engine be kept sufficiently warm, then it was found that the same miles per gallon could be obtained with a 30 per cent. water emulsion as could be obtained with neat petrol, but as already stated there are certain factors which must be considered before one can draw any conclusions from the above statement. If any member is interested in this particular phase of the work, the writer would be pleased to furnish further detail.

(ii.) *Intensive Mixing of Liquids.*—Passing on to the next general use of the mill we come to the intensive mixing of liquids and solids and the blending of difficultly miscible constituents in various chemical preparations.

The enormous forces set up in the film between the confines of the rotor and stator in the mill make it quite obvious that the churning effect makes the mill ideal for the uses above defined, and it is hardly necessary to comment on its possibilities in this direction.

(iii.) *Comminution of Solids.*—Regarding the minute disintegration of solids we are limited here to certain degrees of hardness because we can only break up particles with the disintegrating forces which we have within the film of liquid, and these forces are naturally controlled by the thickness of the film and by the peripheral speed of the mill. The practical limit in the first case is about three-thousandths of an inch, and this limit is controlled by the limits of accuracy with which the machine can be made and by the slight wear which is likely to take place in the bearings, but more particularly by the distortion of the castings.

These defects could be overcome but the cost of the machine would be prohibitive. The peripheral speed of the machine is limited by the quality of modern bearings on the one hand and the capability of constructing a rotor to withstand the tremendous centrifugal forces which develop at high speed. Here again we could exceed our present limits a very great deal, but the cost of manufacture prohibits one from going very far except for experimental purposes.

Experience shows that one can never predict with certainty whether a certain solid substance can be satisfactorily dealt with as far as fine disintegration is concerned, and the ordinary scale of hardness is a very little guide.

A material like sulphur which is fairly soft can be disintegrated to a stage where it will pass through an ordinary filter paper. In most cases where one is attempting to obtain a very fine suspension of a solid matter it is necessary to prevent flocking by the addition of some protective colloid, and the nature of this material varies according to the material used. In the case of sulphur a protective in the nature of a gum or glue is found to be very satisfactory, but for some purposes an inert protective such as magnesium hydroxide gel may be used with advantage.

Sulphur in a finely divided state is finding an increasing market as an insecticide, as a constituent of sheep dips, as a material to replace flowers of sulphur in sulphur soaps, and is a means of obtaining a colloidal sulphur bath in the home.

A good deal of competition is going on in America between people who are making such sulphur mechanically and others who are making a similar product chemically.

Material like flake mica can be reduced to a fairly fine state of subdivision without the use of any stabiliser whatever and one can start with material in small flakes about ½ in. in diameter. Such products are used in the printing of wall paper.

Attempts are being made at the present time to utilise a suspension of iron oxide in the purification of gas, but while a satisfactory suspension can be obtained difficulties have arisen in the re-oxidation of the material after the gas has been purified.

The preparation of certain insecticides which consist of a finely divided suspension of lead arsenate, lead chromate, etc., can very readily be made in the mill, and for such fine suspensions we utilise a speed of 5000 revolutions per minute and a clearance of about four-thousandths of an inch. The material can be circulated through the mill by joining the suction and delivery together and a very intensive treatment thus obtained.

Many organic chemicals which are extremely difficult to grind owing to their greasy nature may be very simply treated in the Premier Mill. There is another class of bodies which is almost impossible to grind owing to the difficulty of wetting with water. Certain dyestuffs in the dry form have been submitted to the writer which could not be wetted after stirring for a very considerable time ; in fact, if one attempted to stir them into water the agitation simply caused the dyestuff to float away as a dust. In such cases we have simply fed the powder and liquid into the mill direct when wetting has been obtained immediately.

Considerable success has been obtained in the grinding of certain dyestuffs in water, and in some cases the resultant material has been a slimy paste. Even with dyestuffs one finds that one can never predict what success one is likely to have.

It is not always permissible to use any protective, so that failure often occurs in this direction for reasons which we cannot control.

(iv.) *Extraction.*—The extraction of certain constituents from various liquids can readily be carried out with the aid of the Premier Mill, and as an example we might quote the extraction of tar acids from tar oil.

In the ordinary way this is done in a mixer by means of caustic soda, mixing taking place until the tar acids have all combined with the soda. The mix is then allowed to settle and the two layers separated.

Instead of using this type of mixer one can advantageously replace it with a mill of the type of the

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Premier. The caustic soda and tar oil are then run simultaneously into the mill and the rates of flow adjusted by flow meters in order that the proper quantities are permitted to react. The reaction is instantaneous in the mill and the resultant product can then be run into the centre separating column filled with packing. The carbolate and cresylate sink to the bottom, while the washed oil floats to the top. Both products can then be run off simultaneously and continuously.

(v) *Extraction of Fibrous Tissues.*—Passing on to the extraction of fibrous tissues we would make the following remarks.

It has been found by experiment that the mill does not disintegrate to any marked extent, vegetable products such as barks, roots or leaves. Advantage has been taken of this fact to extract the active principles from various natural products by mixing them with suitable solvents and passing them through the mill. The function performed by the mill appears to be one of hydraulic maceration and most satisfactory results have been obtained.

For example, the following is an account of the results obtained in dealing with cinchona bark. The types of bark used were Carthagena Flat, Carthagena Quill, Guayaquil and Pitayo.

The treatment and results were in all cases identical. The selected bark was ground to 40 mesh and mixed with 10 per cent. lime. The product when dried is ready for treatment.

The prepared material was mixed with 10 times its weight of cold solvent (Heavy Naphtha, 95 per cent. ; Fusel Oil, 5 per cent., gave very satisfactory results, although many other solvents can be used) and passed once through the mill.

The resultant mixture is easily filtered and the solvent has picked up very little colouring matter. The treated bark, on washing, in all cases failed, on suitable treatment, to yield sufficient alkaloid to react with Majers Reagent.

The extraction might be said to be absolute.

The total alkaloids in the limed barks treated by this process varied between 2·5 per cent. and 3 per cent., and the mill can handle from 15 cwt.—20 cwt. of bark per hour.

(vi.) *Manufacture of Paints, etc.*—The next field that we come to is that of the manufacture of certain paints and enamels.

In such cases as the manufacture of a white enamel, in which the pigment needs very little actual grinding, but where it is essential to thoroughly wet and incorporate the varnish into the pigment, the mill appears to give very satisfactory results, and a large number of machines are used for this purpose.

The grinding capabilities of the mill limits its use to certain pigments, but it is found possible to manufacture a considerable number of these commercial paints and enamels with the use of the mill alone without resorting to orthodox paint machinery in any form.

For certain cheap commercial paints which are required in large quantity, the high output of the mill is a very big attraction, and for a good many of these products a satisfactory result can be obtained at a speed of 100 gallons per hour, it being necessary merely to mix the pigments and liquid medium in a rough mixer beforehand.

In the case of enamels a speed of from 25 to 60 gallons per hour can be obtained, depending upon the quality of the product required.

The different phases in paint work are so many and varied that it is not possible at this juncture to do more than indicate the line of application.

(vii) *Regeneration of Milk.*—An extremely useful work which is perfectly carried out by means of the Colloid Mill is that of the regeneration of milk from milk powders and the production of synthetic cream from butter and milk powder. The use of this regenerated cream is slowly growing in this country. In the United States of America enormous quantities are used, and the Premier Mill finds a ready market in that country for the homogenisation of ice cream paste and the production of synthetic cream.

The smallest type of Colloid Mill is generally regarded as a laboratory type machine, but this will readily make 30 to 40 gallons of cream per hour, and utilises only about 1½ horse power. The constituents are heated to about 55° C. in a mixing tank, and are run through the mill at a clearance of about ten-thousandths of an inch. The resulting product is cooled as it leaves the mill.

The material is perfectly homogenised, and in the opinion of those experienced in this work the result is extremely good.

Combining all the different effects outlined above, one can readily see that there are hundreds of applications in the chemical industry which have scarcely been touched up to the moment.

In the manufacture of pharmaceutical preparations alone, the mill has hundreds of applications, and this fact is rapidly being realised in the United States, and more slowly in this country.

The study of the control of emulsions is receiving considerable attention from those interested in this particular mill, and it is hoped shortly to be able to guarantee the stability of almost any emulsion for an indefinite period, but to this end chemists will realise that stabilisers must be found that do not decompose in dilute solution.

It is well known, of course, that the stability of an emulsion or a suspension depends entirely on certain electrical factors, and the problem is entirely a physico-chemical one.

The state of this research at the present time is such that it indicates that the control of an emulsion will be carried out almost entirely by the accurate adjustment of the hydrogen ion concentration of the medium.